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Journal

—OF THE—

American Ceramic Society

A Monthly Journal devoted to the Arts and Sciences Related to the Silicate Industries

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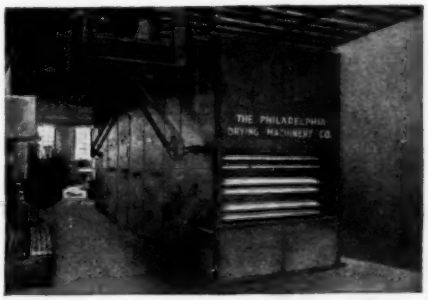

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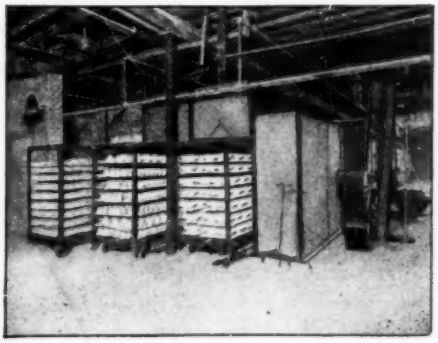

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A monthly Journal devoted to the arts and sciences related to the silicate industries.

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Vol. 4 **November, 1921** **No. 11**

EDITORIAL

THE EXECUTIVE CONFERENCE

The Executive Conference held at the Hotel Commodore, New York City, Sept. 15th and 17th, was attended by approximately fifty past or present officers, members of committees, etc.

The discussion covered an outline of topics that was prepared by the Organizing Secretary, Mr. Purdy, constituting a survey of the problems pertaining to the Society as a whole, and more particularly those pertaining to the efficient functioning of the Sections and Divisions.

As a result of the discussions, certain changes in the constitution and in the by-laws were proposed, which were read at the regular meeting of the Society, held on Sept. 16th, Ceramic Day, at the Chemical Exposition. These will be submitted to the membership for vote.

Further, a number of recommendations were made to the board of trustees for their consideration and action. The results of the final action on the various proposals and recommendations will be published as soon as the vote has been completed, which should be at an early date.

It is beyond question that the Society is on the threshold of a greatly extended program in accordance with its original and reaffirmed purpose, *viz.*, "To advance the ceramic arts and

sciences." In the interpretation of this purpose a closer contact with the ceramic manufacturer is being developed. In its finality, the activities of the Society redound to the economic benefit of the manufacturer in particular and to the economic and aesthetic benefit of humanity in general.

This program of larger service will naturally require more money. The board of trustees has inaugurated the agencies of this service with the conviction that all ceramic entities, industrial associations, corporations, and individuals, will be pleased to contribute proportionately to the financing of the project and will be assured that every dollar so invested has been most advantageously placed.

It is the duty and personal privilege of each member of the Society to give his quota of the larger service and to help solicit the needed financial support.

F. K. PENCE.

TO AUTHORS OF PAPERS

Authors' manuscripts and original drawings are preserved in this office for twelve months following date of publication, after which they are destroyed. Authors desiring any of this material returned to them should notify the Editor's office within the twelve month period.

ORIGINAL PAPERS AND DISCUSSIONS

POSSIBILITIES OF TERRA COTTA CASTING

By R. F. Geller[1]

ABSTRACT

Experimental method.—The casting properties, viscosity, specific gravity and drying behavior of three kaolins, two ball clays, one fire-clay and four representative commercial bodies, together with terra cotta clays, were studied. The clays and bodies were treated with varying amounts of salts and cast in the form of small ashlars.

Conclusions.—Kaolins have an open structure but do not develop strength on drying. Bonding properties are furnished by ball clays which, however, tend to seal against the mold. Commercial terra cotta bodies tested do not possess good casting properties but can be modified through a study of the component clays. Mechanical difficulties attending the casting of terra cotta render the feasibility of the process doubtful at the present time.

Introduction

A general tendency toward improvement of terra cotta and its method of manufacture has made it desirable to study the possibilities of the casting process for the formation of clay ware as applied to this important industry. The present work consists of a preliminary study of the casting behavior of three kaolins, two ball clays and one fire-clay. This is followed by a number of laboratory tests in which representative commercial terra cotta bodies were treated with different and varying amounts of salts and the effect on their casting properties, viscosity, "specific gravity" and drying behavior noted.

The following commercial bodies and clays were tested: Two New Jersey bodies, as well as the clays used in their manufacture; one Indiana body; and one body used in California. In

[1] Received July 18, 1921. Published by permission of the Director, Bureau of Standards.

the preliminary work, washed North Carolina, Georgia and Florida kaolins, Tennessee and Kentucky ball clays and an Arkansas fire-clay were used. Whenever necessary, the clays were dried and crushed to pass a twenty mesh sieve before placing in the blunger where the body was thoroughly mixed to a stiff mud consistency. When barium carbonate was used to react with sulfate present as an impurity in the clay, it was added as a powder during this stage. Mixing was then continued for an hour before addition of the deflocculating agent. After the reaction with the salts and required additions of water had brought the mixture to the proper consistency it was poured into plaster molds to cast. The slip was considered right for casting when it would just flow from a container. "Specific gravity" determinations were made by simply obtaining the weight of 100 cc. of the slip, and comparing it with the weight of 100 cc. of water and the viscosity was measured by means of a viscosimeter similar to the one used by Bleininger.[1] Linear drying shrinkage was obtained on the cast piece itself, and small samples poured in a plaster mold were used for the water-of-plasticity determinations. The grog used in the testing of the kaolins, ball clays and fire-clay consisted of ground white ware bisque. Fifty per cent of this grog passed the 15 mesh sieve and was retained on the 40 mesh and the balance passed through the 40 mesh. The commercial clays were mixed in equal proportions with grog furnished by the manufacturer. All the casting tabulated in table No. 1 was done in plaster molds designed to form an ashlar $6 \times 6 \times 4$ inches, and, in order to obtain comparative results, an equal time was allowed before removing specimens from the mold. In certain cases where it was obvious that the piece would not bear removing the time was lengthened as noted.

Considering the first ten experiments in the table, we see that the kaolins in no case produce a good wall, although the specific gravity of the slip and the thickness of the cast wall are satisfactory. The kaolins as a class produce a slip which is heavy, will not flow in the viscosimeter and yet is short. It has none of that stickiness which so characterizes a glass pot casting slip

[1] Bleininger, A. V., "Notes on Castings," *Trans. Am. Ceram. Soc.*, **17**, 331 (1915).

and when poured seems to slide over itself rather than string out and flow, as flowing is ordinarily thought of. The walls have no strength and either slump or crack in drying.

With 0.1 per cent salts, the fire-clay produces a slip similar to that of a kaolin in the way it casts, but it also has considerable gumminess. With 0.2 per cent salts the gumminess increases and the specific gravity is raised over eight per cent. The slip now shows a tendency to form a dense wall against the sides of the mold. This wall, which is usually about one-eighth inch thick at the maximum and will form in about ten minutes, seems to act as a seal which retards and even stops further casting through absorption of water by the plaster.

With the ball-clays, which contain large amounts of colloidal matter and are very fine grained, the greatest amount of reaction with salts is obtained. The ball-clay slips are of high specific gravity and extremely sticky. They are quite mobile during, and immediately after, agitation but set quickly to a "soft mud" consistency. The Tennessee ball-clay forms a seal almost immediately after being poured and, although the entire mass becomes thick, the actual cast wall does not exceed one-fourth inch after two hours' time. The Kentucky ball-clay has less tendency to seal but the wall formed is weak.

It would seem from these results that the more open structure of the kaolins is a desirable feature in casting in order that passage ways may be kept open through the wall already cast to facilitate the absorption of further amounts of water by the mold. This is also shown by the casting behavior of the coarser grained Kentucky ball-clay. On the other hand, colloid matter either organic or inorganic is necessary as a bonding element. This colloidal material is carried in super abundance by the ball clays and it would seem logical then to so blend a "fat" clay with one lean in colloids as to procure a body of the desired quality. Since the material most widely used in making terra cotta contains a semi-fire clay, one would expect it to vary from a fine grained and very plastic body to one of coarse grain and little plasticity. It was, therefore, determined to study the clays used in this industry individually in order that, their properties being known, they might be blended to produce a mixture suitable for casting.

Clay No. 1 of body A cast satisfactorily with every combination of salts. This clay is fine grained and exceedingly plastic. When using commercial sodium silicate (water glass) a viscous slip is formed which seals in casting and in every way resembles that of a ball-clay. This clay is interesting because of its wide range of reactions to various combinations of salts. Using barium carbonate and sodium silicate, the slip has a high viscosity. When sodium carbonate replaces the sodium silicate the gumminess is reduced, and this becomes even more noticeable when the barium carbonate treatment is omitted. When the soluble barium chloride was substituted for the carbonate, sodium silicate being used as the deflocculating agent, the slip formed had no strength. Lastly, when the barium chloride and sodium carbonate combination was used, the resultant slip had no resemblance to the first obtained but appeared and cast up like a kaolin.

Clay No. 2 of the same body is a mixture of what appears to be a ball-clay and a coarse grained fire-clay high in ferruginous impurities, together with decayed organic matter. As shown in test No. 19 a good wall is cast on addition of 0.4 per cent of sodium silicate, which, however, causes efflorescence on drying. It may be said that this clay had no tendency to seal but formed a wall of insufficient strength.

Clay No. 3 is a mixture resembling the preceding one but having a comparatively small amount of the fine grained material and considerably more of the coarse grained clay. In contrast to body No. 1 this mixture developed the best qualities without the use of the barium salt, but the walls in no test developed satisfactory strength. On standing this slip becomes thinner instead of setting to the "gel" state, as was found to be the case with every other clay tested. The reason for this phenomenon is not evident from the data at hand.

Body No. 4, a mixture of what resembles an impure and a comparatively pure fire-clay, has considerable plasticity and a coarse grained structure. With 0.2 per cent of sodium silicate, the slip is "short" similar to a kaolin and when the salts are increased to 0.3 per cent a slip is formed which sets quickly when agitation ceases but does not form a seal in the mold. The wall,

TABLE I

Test No.	Materials used	0.1% $BaCO_3$	Salts used in per cent of dry body*[2]	Max. wall cast in inches	Time required to set, in hrs.	Time required to remove from mold, in hrs.	Quality of wall	"Specific gravity"	Viscosity*[1]	Water of plasticity %	Dry shrinkage: dried at 110°C; per cent of wet length	Remarks
1	N. C. kaolin 50% grog	no	01. M	1	1½	3	poor	1.85	—	——	4.0	Slip would not run. Not gummy. Efflorescent, cracked in drying.
2	Ga. kaolin	no	0.1 M	¾	2	3	poor	1.80	M	——	5.0	Slip would not run. Not gummy.
3	Fla. kaolin 50% grog	no	0.1 M	1	2½	3½	poor	1.54	L	43.7	7.5	Slip would not run. Not gummy.
4	Tenn. ball 50% grog	no	0.1 M	½	2	3	medium	1.82	H	26.8	5.5	
5	Discarded											
6	Ky. ball 50% grog	no	0.1 M	⅞	2	3½	poor	1.70	M	32.5	6.0	
7	Ark. F. C. 50% grog	no	0.1 M	¾	1⅔	3½	poor	1.70	—	32.0	4.2	Sets up like a kaolin. Walls cracked in drying.
8	Ark. F. C. 50% grog	no	0.2 M	¾	1¾	3	medium	1.85	M	23.9	6.5	Entire mass sets. Walls soft but did not slump.
9	Ky. ball	no	0.2 M	⅝	1¾	3	poor	1.80	M	26.0	4.5	
10	Fla. kaolin	no	0.2 M	¾	2	3	poor	1.64	—	35.8	6.5	Slip thin but not viscous. Efflorescent and cracked in drying.

*[1] H: high viscosity; M: medium; L: low.

*[2] Salts used—M: mixed salts—4 parts Na_2SiO_3 to 3 parts Na_2CO_3; CO_3: Na_2CO_3; SiO_3: Na_2SiO_3.

TABLE I (*continued*)

Test No.	Materials used	0.1% $BaCO_3$	Salts used in per cent of dry body*[2]	Max. wall cast in inches	Time required to set, in hrs.	Time required to remove from mold, in hrs.	Quality of wall	"Specific gravity"	Viscosity*[1]	Water of plasticity %	Dry shrinkage: dried at 110°C.; per cent of wet length	Remarks
11	Body A[1] Clay No. 1 and grog	no	M	5/8	2	2½	poor	1.74	M	29.2	4.0	Efflorescence.
12	do.	no	0.2 CO_3	11/16	2	—	poor	1.80	M	28.4	4.5	Cracked in drying. Left in mold over night.
13	do.	0.1 $BaCl_2$	0.2CO_3	¾	3	—	poor	1.70	—	33.8	4.5	Slip more like a kaolin. No gumminess.
14	Discarded											
15	Clay No. 1 and grog	yes	0.2 SiO_2	5/16	1½	—	medium	1.87	H	23.6	4.0	Left in mold over night. Very viscous.
16	do.	yes	0.2 CO_3	½	2	—	poor	1.81	H	28.7	7.5	do.
17	do.	0.1 $BaCl_2$	0.1 SiO_3	⅞	3	4	medium	1.64	L	38.5	6.5	Walls appeared fairly strong but broke in removing. Mass in center remained thin.
18	Clay No. 2 and grog	yes	0.2 SiO_3	¾	3	3½	poor	1.64	L	38.7	5.0	Sandy clay. Acts like a kaolin.
19	do.	yes	0.4 SiO_3	1	2	5	good	1.80	H	28.8	4.0	Good wall but efflorescent in drying.
20	do.	yes	0.4 M	11/16	2	3	poor	1.76	M	30.3	—	
21	do.	yes	0.4 M	1	5	6	poor	1.61	M	37.8	5.0	Walls did not break but slumped.
22	Clay No. 3 and grog	yes	0.2 SiO_3	½	2	3	medium	1.92	H	20.6	3.0	½" set up but 1" could be left.
23	do.	yes	0.2 CO_3	9/16	2	3½	poor	1.82	L	30.3	4.0	Wall had actually set up 9/16" in 1 hr. Slip in center more watery after standing.
24	do.	no	0.2 M	1	2	3½	medium	1.86	M	27.7	5.0	Slight efflorescence.

[1] Clays No. 1, 2, 3 and 4 are those composing Body A

TABLE I (*continued*)

Test No.	Materials used	0.1% $BaCO_3$	Salts used in percent of dry body*2	Max. wall cast in inches	Time required to set, in hrs.	Time required to remove from mold, in hrs.	Quality of wall	"Specific gravity"	Viscosity*1	Water of plasticity %	Dry shrinkage: dried at 110°C; per cent of wet length	Remarks
25	Body A Clay No. 3 and grog	yes	0.2 M	¾	2	3	poor	1.88	M	23.5	4.0	Center more water after standing.
26	Clay No. 4 and grog	yes	0.2 SiO_3	⅞	2½	—	poor	1.74	—	31.0	5.0	Slip like kaolin mix. Left in mold over night.
27	do.	yes	0.3 SiO_3	1¼	3	4	good	1.87	H	25.4	6.0	Sets up well but cracked in drying.
28	do.	yes	0.3 M	1	3	6	medium	1.84	—	26.9	4.5	
29	12½% Clay No. 1 37½% Clay No. 4 and 50% grog	yes	0.25 SiO_3	1¼	2	4	good	1.86	H	25.8	5.5	Wall good. Slip set quickly.
30	50% Clay No. 4 and grog 50% Body D	yes	0.25 SiO_3	1	2	3¼	medium	1.86	H	25.9	4.5	Cracked in drying.
31	25% Body B 75% Clay No. 4 and grog	yes	0.25 SiO_3	1	3	3½	medium	1.91	M	23.2	5.0	Only ½" actually cast. Walls did not slump.
32	50% Body D 50% Clay No. 4 and grog	yes	0.2 M	11/16	2	3	poor	1.75	—	33.8	5.5	Acts like kaolin mix. Slip not viscous, acts like kaolin.

TABLE I (*continued*)

Test No.	Materials used	0.1% $BaCO_3$	Salts used in per cent of dry body*[3]	Max. wall cast in inches	Time required to set, in hrs.	Time required to remove from mold, in hrs.	Quality of wall	"Specific gravity"	Viscosity*[1]	Water of plasticity %	Dry shrinkage; dried at 110°C; per cent of wet length	Remarks
33	25% Body D 75% Clay No. 4 and grog	yes	0.2 M	1	3	3	poor	1.80	L	30.4	—	Slip runs easily, does not string.
34	25% Body D 75% Clay No. 4 and grog	yes	0.2 M	1	2	3	medium	1.83	L	27.3	5.0	Slip apparently had a good casting consistency.
35	Body B	yes	M	3/4	2	3	poor	1.85	H	26.1	5.5	Tends to seal.
36	do.	yes	CO_3	5/8	2	—	poor	1.67	L	37.4	6.5	Set in mold over night. Slip not gummy.
37	do.	yes	0.3%	7/16	1	—		1.73	M	33.3	—	Tends to seal.
38	Body C	yes	M	3/8	2		poor	1.90	H	22.1	7.5	Tends to seal. Left in mold over night. Entire mass thick gum.
39	Body D	yes	M	7/16	2	4	medium	1.76	M	30.3	7.0	Tends to seal. Walls fairly strong considering thickness.
40	Body B 25% Clay 1 25% Clay 2 50% grog A	yes	0.25% M	5/8	2	—	poor	1.91	M	24.2	4.5	Tends to seal. Left in mold over night. Sets quickly.
41	Body B 25% Clay 3 25% Clay 4 50% grog A	yes	0.25% M	1/2	2			1.92	M	21.6		Slip does not set up.
42	Body C	yes	CO_3	1/2	1½	—	poor	1.81	—	29.6	5.5	Left in mold over night. One in. wall left but only ½" firm. Does not seem to set up.

to all appearances strong, sets up in three hours to the required thickness. The shrinkage, six per cent, is comparatively large, however, and the piece cracks in drying. Since the clay is satisfactory in every way but the drying behavior, it was decided to add some of the clay No. 1 in order to obtain the necessary bond. As shown in test 29, a body was prepared containing 12½ per cent clay No. 1, 37½ per cent clay No. 4 and 50 per cent grog. With 0.25 per cent of sodium silicate, the resultant slip formed a wall 1¼ inches thick and very strong, in two hours' time. The piece removed easily from the mold and did not fail in drying.

Body B, as the results of test 35, 36, and 37 show, was not adapted to casting without modification. The mixture, which apparently had a high bond clay content and a low percentage of grog, was very plastic and would seal in the mold. The individual clays of this body were obtained, but not in sufficient quantity to make the desired number of tests. Clay No. 1 was to all appearances a ball-clay, clay No. 2 a fine grained plastic clay almost white in color, and clays No. 3 and No. 4 had the properties of semi-fire clays. From the two preliminary tests which it was possible to make with the small samples furnished, shown in tests 40 and 41, it would seem that a small proportion of clay No. 1 blended with either or both of the fire clays would produce a slip favorable for casting.

Body C, representing the mid-western states, formed a slip too high in viscosity for casting. It was mixed with a lean clay, (see test No. 31) in the ratio of one to three but even then the tendency to seal was too great and only one-half inch of wall was formed after a reasonable length of time. The strength, however, was very good and a study of the individual clays would no doubt result in the making of a desirable body.

Body D, from California, was of a buff color and contained a large amount of comparatively coarse inert material. The casting test of this body was not promising. There was a tendency to seal without the mass setting-up to any stiffness. When blended in equal parts with a lean clay (see test 30) the casting proceeded normally but the structure formed was not sufficiently strong to withstand drying strains. It would seem that a coarse grained plastic clay, such as a fire-clay, would help the general

qualities of this body for casting. Several tests which have been suggested were not carried out in this work because of lack of materials and time, but sufficient data are here shown to indicate that, based on a study of the several clays, other combinations suitable for casting besides that of test 29, can be developed.

The main difficulty encountered in this work lies in the handling of the slip. In the making of glass pots a large amount of slip is concentrated in the making of one piece, and the points where casting is done are proportionately fewer and nearer to the mixer than in the case of a plant for the manufacture of terra cotta. There the pieces are small and numerous and consequently would be scattered over considerable area. To carry the slip to the molds in large containers would necessitate an elaborate system of over-head cranes and to carry the molds to the mixer would mean a carrier system no less expensive. The most convenient system would, of course, be to pump the material. When one considers, however, the viscosity of the mass to be handled as well as the fact that it tends to set, after the nature of colloids, as soon as movement due to externally applied pressure is stopped, it immediately becomes evident that the size of piping and pumping force required would make the cost of the equipment practically prohibitive.

Now, presupposing that the above difficulties have been overcome and that the slip is in the mold, we have the following conditions to be met. Since no cores are used, that portion of the slip in the center of the mold must be removed after the walls have been cast. With the making of pottery this superfluous matter is simply poured out, leaving a uniform shell of material against the mold. In the casting of heavier ware, with a mix containing a large proportion of coarse grog and in which the water content must be kept as low as possible, the slip, originally sluggish, has become "livery" throughout by the time the casting is complete. Consequently the center slip can not be poured out and can be even less readily pumped. Also, there is no sharp demarcation showing the cast portion, so that the bottom and sides of the piece must be worked to the desired uniformity by hand. It is possible of course to obtain a thin

slip and one which will not set to a "livery" mass, but in none of the present tests did such a slip cast a wall of any strength. Having removed the mass from the center of the mold by dipping or some other suitable method, it must be returned to the blunger and agitated until it again becomes uniform and free of lumps. A few trials were made in which this slip was put directly into a second mold, and it was found that the casting properties had been impaired, probably through a setting of part of the colloid. It has been found in the casting of glass pots that it is necessary to treat a slip, which has once set, with a small addition of salts to restore its original ability to cast and this would in all probability hold for the terra cotta slips. This treatment would therefore be necessary in connection with the blunging mentioned above.

The question of webs is also of importance. In casting without a core it would of course be necessary to place these partitions in the piece after removal of the superfluous slip. The webs, in all probability machine-made, would require a very careful control of composition and water content in order to insure shrinkage in drying and burning equal to that of the cast walls. The relative compositions of cast and machine-made bodies could only be determined by individual experimentation.

Casting without a core, the following three conditions have then been found to cause unusual difficulties and expense:

a. The transportation of the slip to the mold.
b. The removal of the superfluous slip from the mold.
c. The treatment of this slip to render it again suitable for casting.

To cast with a core would of course entail an additional cost for material and manufacture but to offset this there are the following savings:

a. Depending on the size of the piece, each mold would require the transportation of 40 to 60 per cent less slip in the first process.
b. There would be no superfluous slip to remove from each mold.
c. There being no waste material, it would not be necessary to transport such material back to the blunger and prepare for recasting.

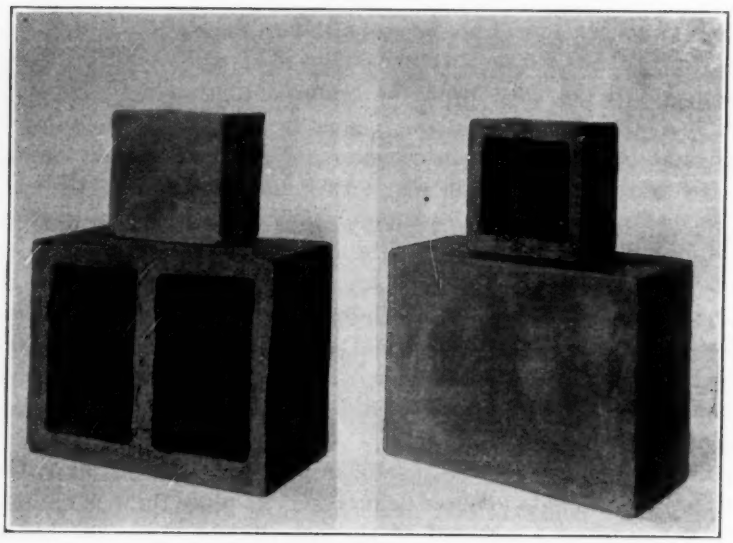


FIG. 1.

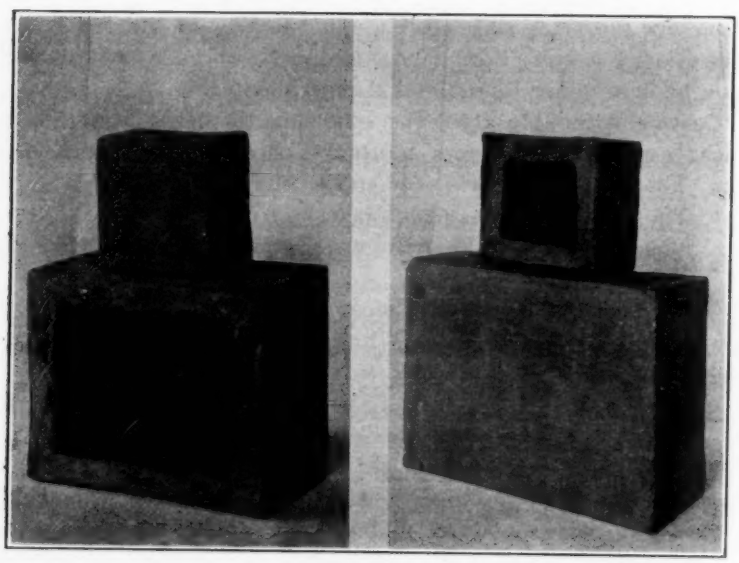


FIG. 2.

d. The same cores could be used for all pieces of approximately the same size and varying only in the different decorative designs.

In order to investigate the feasibility of casting with cores, a few ashlars with approximately one inch thick walls were cast. The specimens were photographed and are shown in figure 1. The test pieces obtained in experiments 19 and 29 are shown in figure 2, together with a larger specimen ($12 \times 9 \times 6$ inches) cast without a core and using the body given in test 29.

From the data as presented in this paper, it would seem that the following conclusions might be drawn concerning the casting properties of kaolins, ball-clays, fire-clays and terra cotta bodies:

a. The open structure of kaolins is a desirable feature in casting but they do not possess sufficient bonding material.

b. The ball clays have sufficient bond, but form a skin against the mold which is too dense to allow the further absorption of water from the slip.

c. Fire clays may have the general casting properties of either a kaolin or the ball clay, depending on their structure and on their potential casting properties due to the salts which they contain as impurities.

d. Commercial terra cotta bodies tested in this work do not possess good casting properties, but may be adapted to this work through a study of their component clays.

e. The mechanical difficulties attending the casting of terra cotta render the feasibility of the process doubtful at the present time.

NOTE ON THE HARDNESS OF GLAZES[1]

By George Blumenthal, Jr.

ABSTRACT

Method of measurement.—The apparatus used (figure 1) consisted of a hardened tungsten-steel point which was allowed to bear on the glaze surface for 3 min. under a pressure of 50 lbs. The hardness was then computed from the dimensions of the resulting indentation. Results were reproducable to about 5 per cent. (See table III.) The presence of ridges and bubbles in the glaze was the principal cause of discordant readings.

Results and conclusions.—The enamels show the lowest and the porcelain glazes the greatest hardness, while the whiteware glazes occupy an intermediate position. The difference in hardness between the enamels and the whiteware glazes is quite marked. There is also a rather well defined difference between the whiteware and the porcelain glazes, but not a sharp separation between the lower and the higher fired porcelain glazes. Enough evidence has been accumulated to show that increased firing of the same glaze will increase the hardness. It appears also that increase in the alumina content brings about greater hardness.

Method

Manufacturers of white ware and table porcelain endeavor to produce as hard a glaze as possible which will resist the mechanical action of cutlery. This requirement may be considered to be a universal one for all types of glazed ware. Unfortunately, however, the determination of hardness is a difficult task and there is no agreement as to the best method of measuring this physical quality. In general, hardness may be defined as the resistance offered by a body to the penetration of a still harder material. Probably the most satisfactory method is that of Brinell, in which the penetration of a sphere into the material to be tested is measured under a standard which causes the penetration. In the present work an attempt has been made to estimate the hardness of several typical

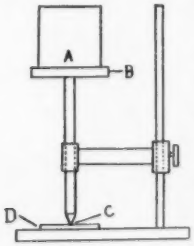


Fig 1.

[1] Received July 1, 1921. Published by permission of the Director, Bureau of Standards.

glazes and enamels by means of an adaptation of the Brinell test. The apparatus employed was very simple and is shown in figure 1. It consisted of a hardened tungsten steel point which was caused to penetrate the surface of the glaze under pressure. This procedure of making the test was a very simple one and consisted in placing a 50 pound weight, A, upon the platform B, and allowing it to remain in position for three minutes, after which time it was removed. The pressure upon the steel point was equivalent to several hundred thousand pounds per square inch. The indentation made in the glazed surface was then measured by means of a micrometer microscope, and several diameters of each impression were measured and a number of impressions made on every trial piece. The mean of the several measurements was taken to be the diameter of the impression. One of the difficulties in making this test was the presence of ridges and bubbles in the glaze, which are the principal causes of many discordant readings.

The computation is based on the relation:[1]

$$t = \frac{D}{2} - \sqrt{\frac{D^2}{4} - \frac{d^2}{4}}$$

where t = depth of indentation, D = diameter of the rounded point, d = diameter of indentation, and p = load. The hardness may then be computed from the relation:[1]

$$H = \frac{p}{\pi tD}$$

The hardness of different whiteware and porcelain glazes was determined as well as that of a number of enamels of the type applied on steel. The whiteware and porcelain glazes were applied upon specimens made in the laboratory. In the whiteware glazes of table II, glazes 1, 2, and 3 have the same molecular formula but different percentages of frit were added in each of the three mentioned.

Results

The observations made in connection with tests on a typical whiteware glaze, fired at cone 4, are given in table I. From

[1] Bureau of Standards, *Tech. Paper*, **11**, "Comparison of Five Methods used to Measure Hardness," p. 5.

TABLE I—CHARACTER OF THE INDENTATIONS ON WHITEWARE GLAZES FIRED TO CONE 4

	Diameter of Impression in mm.	Appearance	Impression
1.	0.424	Hardly any bubbles	Sharp
2.	.460	Few bubbles	Sharp
3.	.464	Many bubbles	Ragged
4.	.490	Many bubbles and fractured at point of contact	Ragged & chipped
5.	.472	Fractured at point of contact	Ragged & chipped
6.	.478	No bubbles—crazed	Ragged
7.	.412	Few bubbles	Sharp
8.	.404	Few bubbles	Sharp
9.	.488	Bubbly, crazed at point of contact	Ragged & chipped
10.	.466	Few bubbles	Sharp
11.	.432	Few bubbles	Sharp
	Same as above at Cone 6		
1.	.356	Few bubbles	Sharp
2.	——	Immature	Ragged
3.	.366	Slightly pitted	Sharp
4.	.372	No bubbles	do.
5.	.398	Few bubbles	do.
6.	.376	Few bubbles	do.
7.	.346	Few bubbles	do.
8.	.366	Slightly pitted	do.
9.	.372	Few bubbles	do.
10.	.354	Crazed when pressure was exerted	do.
11.	.374	Few bubbles	do.

these results it is apparent that the presence of bubbles in the glaze will lower its hardness decidedly. From the appearance of the indentation it seemed that there was an extremely thin but exceedingly hard layer or film on the surface of the glaze, similar to the enamel on teeth. There was a distinct difference between the surface and the material below it, which showed a crushed appearance. If there were a succession of hard layers the steel point would crush through each, leaving a series of ellipses. This was actually found in the case of the harder glazes, but was absent in those of a softer character.

In order to determine the effect of increased firing on the hardness of the whiteware glazes, the latter were applied to whiteware discs and fired to cone 4—4½ and cone 5½—6. In the cone 4 tests, the hardness fluctuated from 248 to 296, although several specimens were entirely out of this range and were discarded as immature. The hardness of the specimens fired to cone 5½ was from 268 to 298. Another set of whiteware glazes were fired at cones 4 and 6 and the hardness found to vary from 214 to 258 at cone 4, and 261 to 300 at cone 6.

In general, firing to higher temperature, therefore, appears to yield harder glazes. The hardness values for the series of white ware glazes are given in table II. Table III shows the hardness

TABLE II—INCREASE OF HARDNESS WITH INCREASE OF TEMPERATURE

	Whiteware Glazes								Cone 4		Cone 6	
	K_2O	Na_2O	CaO	PbO	ZnO	Al_2O_3	B_2O_3	SiO_2	R	H	R	H
1.	0.173	0.032	0.456	0.217	0.122	0.238	0.271	2.30	0.212	246	0.178	249
2.	.173	.032	.456	.217	.122	.238	.271	2.30	.230	226		
3.	.173	.032	.456	.217	.122	.238	.271	2.30	.232	224	.183	284
4.	.173	.139	.343	.219	.124	.240	.278	2.32	.245	214	.186	280
5.	.177	.243	.233	.222	.125	.243	.279	2.35	.236	220	.199	261
6.	.230	.318		.289	.163	.317	.361	3.06	.239	218	.188	276
7.	.266	.211		.334	.189	.366	.417	3.54	.206	253	.173	300
8.	.317	.059		.399	.225	.437	.497	4.22	.202	258	.183	284
9.	.295	.290		.265	.150	.375	.331	2.79	.244	213	.186	280
10.	.368	.182		.288	.162	.452	.359	3.01	.233	224	.177	296
11.	.478	.045		.305	.172	.570	.380	3.05	.216	240	.182	286

R = Radius of indentation in mm.
H = Hardness of glaze.

TABLE III—ILLUSTRATING THE VARIATION OF THE RESULTS USING A SINGLE GLAZE APPLIED TO 29 DIFFERENT WHITEWARE TEST DISKS.

Composition of glaze suitable: CaO 0.55, K_2O 0.25, PbO 0.20, Al_2O_3 0.27, B_2O_3 0.39, SiO_2 2.96.

	Cone 4		Cone 6	
	R	H	R	H
Mean of 29 tests	0.191	272	0.187	280
Average deviation from the mean a.d.	.0068	10	.004	6
Max. dev. from mean	.019	24	.013	18
Percision Measure $\sqrt{\frac{\text{a.d.}}{29}}$	.0013	2.22	.00088	1.33

R = radiation of indentation.
H = hardness of glaze.

of the same whiteware glaze applied to 29 specimens on discs of the same body composition. The specimens were all prepared at the same time. The table shows the range of values that may be encountered in the same glaze on a number of pieces of ware.

The results obtained on porcelain glazes are compiled in table IV, and those for enamels in table V. It is apparent that a distinct

TABLE IV—HARDNESS OF SOME PORCELAIN GLAZES FIRED AT CONE 16

	CaO	K_2O	Al_2O_3	SiO_2	Al_2O_3:SiO_2	R	H	
1.	0.7	0.3	0.30	3.0	1:10	0.146	356	
2.	.7	.3	.375	3.0	1:8	.140	370	
3.	.7	.3	.500	3.0	1:6	.150	346	
4.	.7	.3	.600	3.0	1:5	.156	334	
5.	.7	.3	.750	3.0	1:4	.162	321	
6.	.7	.3	.400	4.0	1:10	.170	306	immature
7.	.7	.3	.500	4.0	1:8	.155	336	
8.	.7	.3	.666	4.0	1:6	.152	342	
9.	.7	.3	.800	4.0	1:5	.147	354	
10.	.7	.3	1.000	4.0	1:4	.149	349	
11.	.7	.3	.500	5.0	1:10	.162	321	
12.	.7	.3	.625	5.0	1:8	.144	361	
13.	.7	.3	.833	5.0	1:6	.142	366	
14.	.7	.3	1.000	5.0	1:5	.151	340	
15.	.7	.3	1.250	5.0	1:4	.153	344	
16.	.7	.3	.700	7.0	1:10	.150	346	
17.	.7	.3	.875	7.0	1:8	.145	362	
18.	.7	.3	1.166	7.0	1:6	.148	350	
19.	.7	.3	1.400	7.0	1:5	.139	372	
20.	.7	.3	1.750	7.0	1:4	.138	374	
21.	.7	.3	.900	9.0	1:10	.155	336	
22.	.7	.3	1.125	9.0	1:8	.160	325	
23.	.7	.3	1.500	9.0	1:6	.158	330	
24.	.7	.3	1.800	9.0	1:5	.148	351	
25.	.7	.3	2.250	9.0	1:4	.147	352	
26.	.7	.3	1.100	11.0	1:10	.144	361	
27.	.7	.3	1.375	11.0	1:8	.143	362	
28.	.7	.3	1.833	11.0	1:6	.151	340	
29.	.7	.3	2.200	11.0	1:5	.138	374	

R = Radius of indentation in mm.
H = Hardness of glaze.

TABLE V—HARDNESS OF SOME TWO-COAT AND GREYWARE ENAMELS

	1	2	3	4	5	6	7	8	9	10
R	0.250	0.252	0.260	0.260	0.258	0.250	0.246	0.250	0.260	0.258
H	208	206	200	200	201	208	210	208	200	201

R = Radius of indentation in mm.
H = Hardness of glaze.

difference in hardness is to be noted. The greatest hardness is shown by the porcelain glazes and the lowest by the enamels. Attention must, however, be called to the fact that the results obtained for one and the same glaze are more or less discordant. The thickness of the glaze is evidently an important factor as well as the varying degree of firing which the specimens may have undergone. It cannot be expected that the results are more than rough approximations. They simply indicate the order of magnitude of the hardness. It is evident that the difficulties in the way of making exact determinations are very great. It is obvious that the chemical composition must have an important bearing on the hardness of the glazes, but no conclusive evidence can be deduced on this point.

One point of interest may be noted, however, namely, that an increase in the alumina content appears to bring about a distinct increase in the hardness of the glaze.

In the following summary table, the maximum and minimum hardness, together with the average hardness of each type of glaze, is given:

		Low	High	Total average
Whiteware Glaze No. 1	Cone 4	213	258	230
	Cone 6	249	300	279
Whiteware Glaze No. 2	Cone 4	248	296	270
	Cone 6	265	298	281
Porcelain Glaze	Cone 16	306	374	348
Two-Coat and Greyware Enamels		200	210	204

A STUDY OF SOME BOND CLAY MIXTURES[1]

By D. H. Fuller

ABSTRACT

In order to determine whether as good results could be obtained from mixtures of American clays as from the Gross Almerode clay in glass pot bodies, two American ball clays were combined in various proportions with two silicious American bond clays and compared with the Gross Almerode clay in bodies containing 50% grog. Physical properties and resistance to glass batch corrosion were determined. It was not found possible to duplicate the physical properties, especially the low water of plasticity and low drying shrinkage of the Gross Almerode body but several of the bodies gave better results as regards resistance to corrosion than the one containing the Gross Almerode clay. In general it was concluded that it is possible by blending American clays to produce a body equal or superior to the Gross Almerode body. More thorough heat treatment of pots or tank blocks in the arches and greater uniformity in temperature during the burning is required.

Introduction

The object of the present work was the study of combinations of aluminous with silicious bond clays, having in mind the utilization of such mixtures as refractories for the glass industry. It is a well-known fact that one of the best clays employed in this connection is the silicious material from Gross Almerode, which combines low drying and firing shrinkage with excellent plasticity and bonding power. An American clay having very similar properties is that from the mine of the Camden Clay and Coal Company, Lester, Arkansas. In addition there are available the more or less silicious pot clays from Missouri, the sandy clays from New Jersey and a considerable number of fire-clays, carrying much free silica, like those from the Ottawa, Ill., district; Mayfield, Ky.; Whitlock, Tenn.; and Warriors' Mark, Penna.

Further knowledge concerning the properties of mixtures of these silicious with the more aluminous bond clays would appear to be desirable, since it would seem from previous work that we must look to such combinations for our best results.

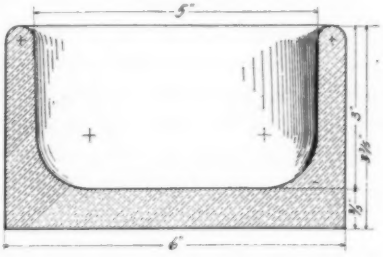
[1] Received September 8, 1921. Published by permission of the Director, Bureau of Standards.

The requirements for bodies of this type are good plasticity and bonding power, a fire shrinkage lower than that obtained by the exclusive use of ball clays, good resistance to corrosion by the glass, and sufficient rigidity under furnace conditions to avoid undue softening and consequent deformation. A pot body as silicious and open as the Gross Almerode is not desirable for all-round work, nor is one as aluminous as the dense plastic clays used in pottery work. A compromise between these two extremes would seem to offer the best solution. The objections to the German clay are its porous structure, which is vigorously though uniformly, attacked by the more fluid glasses, and its sensitiveness to sudden temperature changes. The drawbacks of the aluminous clays are their large firing shrinkage, and a tendency to deform at temperatures somewhat above those of normal operation.

Outline of Work

The experimental work consisted in making the usual determinations of the water of plasticity, drying shrinkage and modulus of rupture of the dried clays and bodies, porosity and shrinkage at different firing temperatures and modulus of rupture of the fired materials. The rate of firing was 20°C per hour. The trials were carried to cones 8 and 14. In addition, the resistance to the action of a corrosive glass was studied, using test crucibles five inches in diameter and three inches deep, shown in figure 1. These were fired to cone 12 and then filled with a raw mixture consisting of 100 parts of sand, 68 of soda ash, 20 of whiting and 2.8 of carbon,[1] and brought to a temperature of 1400°C where they were held for 12 hours. After

Fig. 1



[1] The original glass batch here employed contains salt cake. When this was replaced by soda-ash, the fact that carbon was no longer needed was overlooked.

breaking the cold specimens through the middle, the extent of the corrosion could be observed.

Four clays were introduced in this work, the ball clay No. 5 from Whitlock, Tenn.; the ball clay No. 4 from Mayfield, Ky.; the silicious bond clay from Lester, Ark.; and a saggar clay from Trenton, N. J., known as "Dogtown Clay." The physical properties of the first three clays have been reported upon in previous work,[1] in which they are given as numbers 13, 17 and 23. It may be stated here that the Dogtown Clay was not as silicious as had been expected.

The percentage chemical compositions of these clays were found to be as follows:

	Lester Ark.	Trenton N. J.	Whitlock Tenn.	Mayfield Ky.	Gross Almerode
Silica	72.05	54.13	47.70	53.22	73.08
Alumina	17.21	31.19	33.28	32.36	15.75
Ferric oxide	1.25	2.87	1.21	1.17	0.62
Titanium oxide	Not det.		Not det.		2.10
Lime	0.45	0.21	0.45	0.40	0.54
Magnesia	0.75	1.05	0.75	0.83	0.33
Potash	Not det.		Not det.		0.27
Soda	do.		do.		0.16
Loss on ignition	8.56	10.71	16.25	12.44	7.11

Mixtures were made of each aluminous clay with each of the silicious materials in the proportions of 4:1, 3:2, 2:3 and 1:4. Grog was also prepared consisting of 80 per cent Tennessee ball clay No. 5, 10 per cent of Georgia and 10 per cent of North Carolina kaolin, fired to cone 12. Bodies were made, using 50 per cent of each bond clay mixture to 50 per cent of grog. The crucibles for the slagging tests were made from these bodies and from an additional mixture consisting of 50 per cent of the above grog and 50 per cent of Gross Almerode pot clay. A series of bodies in which the above grog was mixed with each of the four bond clays in the ratio of 50:50 were submitted to the same tests as the mixtures of the blended clays with grog. The compositions of all the mixtures and bodies are given in table I.

[1] *Trans. Am. Ceram. Soc.* **19**, 601 (1917).

TABLE I—COMPOSITIONS OF MIXTURES. PER CENT

No.	Tennessee ball	Kentucky ball	Arkansas	Trenton	Gross Almerode	Grog
A-1	80	..	20	..	..	..
A-2	60	..	40	..	..	..
A-3	40	..	60	..	..	..
A-4	20	..	80	..	..	..
B-1	..	80	20	..	..	..
B-2	..	60	40	..	..	..
B-3	..	40	60	..	..	..
B-4	..	20	80	..	..	..
C-1	80	..	..	20	..	..
C-2	60	..	..	40	..	..
C-3	40	..	..	60	..	..
C-4	20	..	..	80	..	..
D-1	..	80	..	20	..	..
D-2	..	60	..	40	..	..
D-3	..	40	..	60	..	..
D-4	..	20	..	80	..	..
E-1	40	..	10	..	..	50
E-2	30	..	20	..	..	50
E-3	20	..	30	..	..	50
E-4	10	..	40	..	..	50
E-5	..	40	10	..	..	50
E-6	..	30	20	..	..	50
E-7	..	20	30	..	..	50
E-8	..	10	40	..	..	50
E-9	40	..	..	10	..	50
E-10	30	..	..	20	..	50
E-11	20	..	..	30	..	50
E-12	10	..	..	40	..	50
E-13	..	40	..	10	..	50
E-14	..	30	..	20	..	50
E-15	..	20	..	30	..	50
E-16	..	10	..	40	..	50
E-17	50	..	..	..	..	50
E-18	..	50	..	..	..	50
E-19	..	..	50	..	..	50
E-20	..	..	..	50	..	50
E-21	..	..	..	..	50	50
F-1	..	..	..	..	100	..
F-2	100	..	..	..	..	..
F-3	..	100	..	..	..	..
F-4	..	..	100	..	..	..
F-5	..	..	..	100	..	..

Results

Physical Tests.—The results of the tests on the unfired clay and grog mixtures as well as those referring to their transverse strength, when fired to cone 12, are given in table II.

TABLE II—RESULTS OF PHYSICAL TESTS UPON UNFIRED AND FIRED CLAYS

No.	Water per cent	Volume drying shrinkage, per cent	No.	Modulus of rupture, pounds per square inch	
				Dry state	Fired state
A-1	35.83	37.1	E-1	320	2252
A-2	38.49	33.15	E-2	343	2175
A-3	35.93	34.92	E-3	351	1787
A-4	33.35	35.38	E-4	339	2739
B-1	41.33	38.60	E-5	356	2132
B-2	37.38	38.70	E-6	355	2596
B-3	35.98	37.70	E-7	320	2003
B-4	32.25	35.66	E-8	284	1779
C-1	43.72	35.81	E-9	262	2583
C-2	43.88	32.08	E-10	246	2630
C-3	44.53	33.94	E-11	206	2549
C-4	44.07	33.00	E-12	270	2886
D-1	45.70	39.90	E-13	321	2790
D-2	43.90	34.91	E-14	268	2708
D-3	44.35	35.41	E-15	246	3064
D-4	45.73	35.35	E-16	221	2966
F-1	25.27	26.40	E-17	331	2625
			E-18	268	2466
			E-19	421	1473
			E-20	201	2827
			E-21	332	2210

It appears from these results that no combination of these clays possesses as low a water content or drying shrinkage as the Gross Almerode clay, 25.27 and 26.40 per cent, respectively, which, however, is not a fact of great importance for products that must contain from 50 to 60 per cent of grog.

A number of the grog mixtures in the dried state show a higher modulus of rupture than that containing the Gross Almerode clay, especially numbers A-2, A-3, B-1 and B-2. This is still more evident for the strengths in the fired state. The Arkansas clay by itself apparently does not possess a high strength, when fired, and any addition of Tennessee or Kentucky ball clay improves it, although not always in a consistent manner, since,

Fig. 2

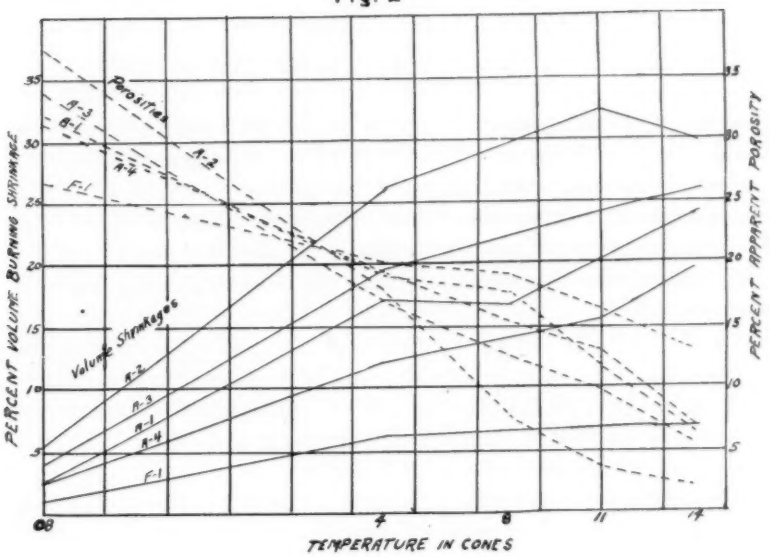
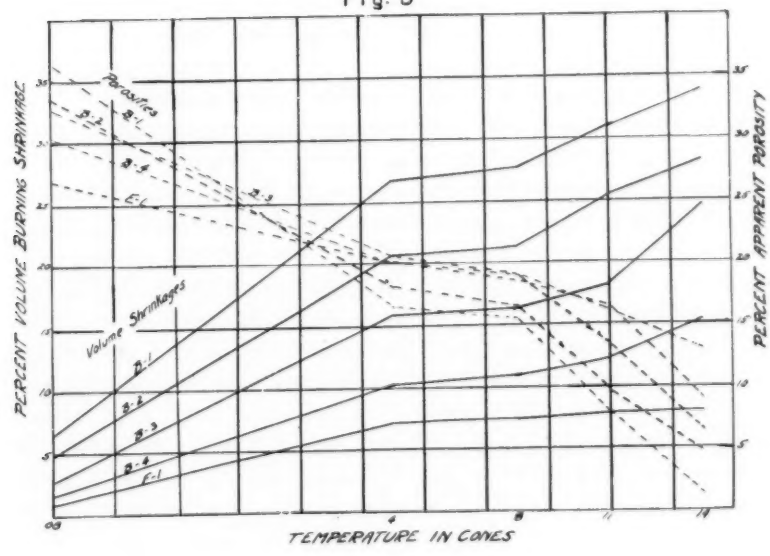

PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
TEMPERATURE IN CONES
Porosities
Volume Shrinkages
A-1
A-2
A-3
A-4
B-1
B-2
B-3
B-4
F-1
08
4
8
11
14

Fig. 3


PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
TEMPERATURE IN CONES
Porosities
Volume Shrinkages
A-1
A-2
A-3
A-4
B-1
B-2
B-3
B-4
F-1
08
4
8
11
14

for instance, a 1:4 mixture of Tennessee ball clay and Arkansas clay shows a higher modulus than with higher amounts of the former.

Firing Behavior.—The volume and porosity changes of the clay mixtures are best studied in groups. Let the combination of Tennessee and Arkansas clays be the first to be examined, using for this purpose the diagram of figure 2. From the fire shrinkage curves, figure 2, it is evident that the lowest and most gradual volume change is that of the Gross Almerode clay F-1. From previous work[1] we know that the Arkansas clay by itself undergoes an even lower fire contraction than the German clay, and we should expect the volume shrinkage of these mixtures to decrease with increasing amounts of the silicious clay. This is true, but the order in which they do so does not conform to the increase in Arkansas clay, since the highest shrinkage is shown by the proportion of 3 Tennessee to 2 silicious material. This discrepancy remains to be explained.

The highest proportion of silicious clay does, however, show the lowest contraction. This particular curve shows an accelerated volume change between the temperatures of cones 11 and 14. Practically the same facts are observed from the porosity curves of figure 2, from which we note also that the density of the clay mass of the mixtures is decidedly greater than that of the Gross Almerode clay.

Mixtures of Kentucky ball clay No. 4 and Arkansas clay, as will be observed from the shrinkage-temperature curves of figure 3, decrease regularly in contraction with the amount of the more silicious material, and a combination in the proportion of 4 Arkansas to 1 Kentucky clay approaches the Gross Almerode clay quite closely. It is not at all unlikely that the mixture B-3, in the proportion of 2 Kentucky to 3 Arkansas clay would be satisfactory all around and overcome the defects of both types of clay. The minimum pre-firing pot arch temperature of this set of combination appears to be cone 4. The porosity and temperature curves, figure 3, give us similar information showing very close agreement in behavior between the mixture B-4 and the Gross Almerode clay and pointing again to the very promising characteristics of mixture B-3.

[1] *Trans. Am. Ceram. Soc.*, **19**, 601 (1917).

With reference to the admixture of the two ball clays with the New Jersey saggar clay, the results obtained show that the choice of the latter material was not a happy one, as it is not as high in silica as would be desirable for this purpose.

This is shown in the curves of figure 4, expressing the temperature-shrinkage relation of blends between the Tennessee and New Jersey clays. The reduction in fire contraction is not great enough to justify the preparation of such mixtures. A similar conclusion must be drawn from the temperature-porosity relation, figure 4, though here C-4 shows up to somewhat better advantage. While in the shrinkage curves the changes between the different proportions of the saggar clay are consistent with the amounts introduced, there is noted an overlapping of the curves at cone 8, which is resolved again into the regular order at cone 11. The dry strength of these mixtures (see E-9, E-10, E-11 and E-12, table II), when made up with 50 per cent of grog, is also below the average.

The results obtained with mixtures of the Kentucky ball and New Jersey sagger clay are similar to those shown in figure 5 and hence need no separate discussion. No significant changes are to be observed, even with larger addition of the saggar clay, and no material beneficial effect is indicated.

Corrosion.—In studying the resistance to corrosion of the different clays and clay mixtures, the Gross Almerode clay, or No. 21, has been used as the basis of comparison. The results follow:

E-1.—Body structure quite dense, white color. Less attack by glass than G. A.; color of glass better than in G. A. pot.

E-2.—Structure more open and attacked by glass like G. A. body, but color much lighter.

E-3.—Glass attacked as with G. A. body, but color much lighter; structure very similar to G. A. body, but somewhat more dense.

E-4.—Very close to G. A. body though of lighter color; slightly denser than G. A. body.

E-5.—Body more porous than G. A. body but of much lighter color. Glass attack very slight.

Fig. 4

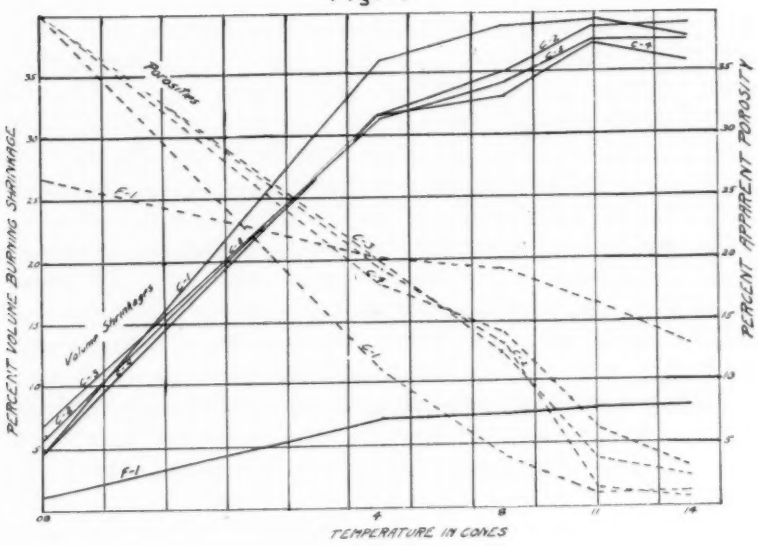

PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
Porosities
Volume Shrinkages
E-1
E-2
E-3
E-4
F-1
08
4
8
11
14
TEMPERATURE IN CONES

Fig. 5

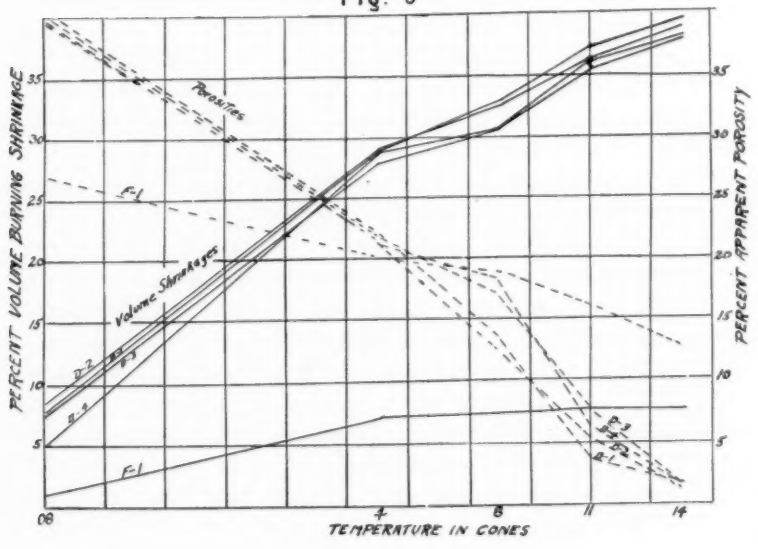

PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
Porosities
Volume Shrinkages
E-1
E-2
E-3
E-4
F-1
08
4
8
11
14
TEMPERATURE IN CONES

Fig. 6

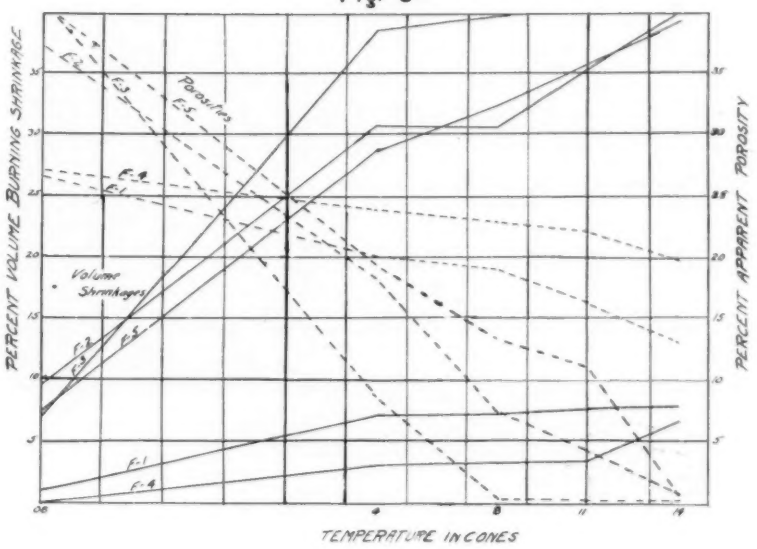

PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
Porosities
Volume Shrinkages
E-1
E-2
E-3
E-4
E-5
TEMPERATURE IN CONES

Fig. 7

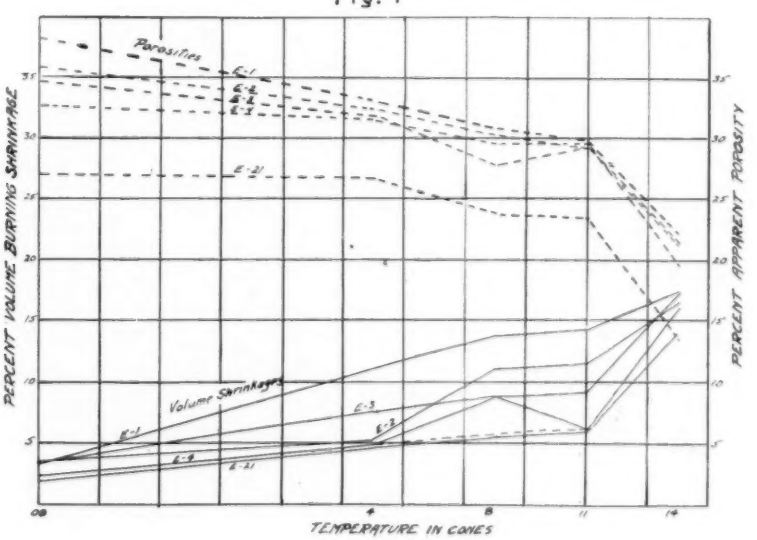

PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
Porosities
Volume Shrinkages
E-1
E-2
E-3
E-4
E-5
E-21
TEMPERATURE IN CONES

Fig. 8

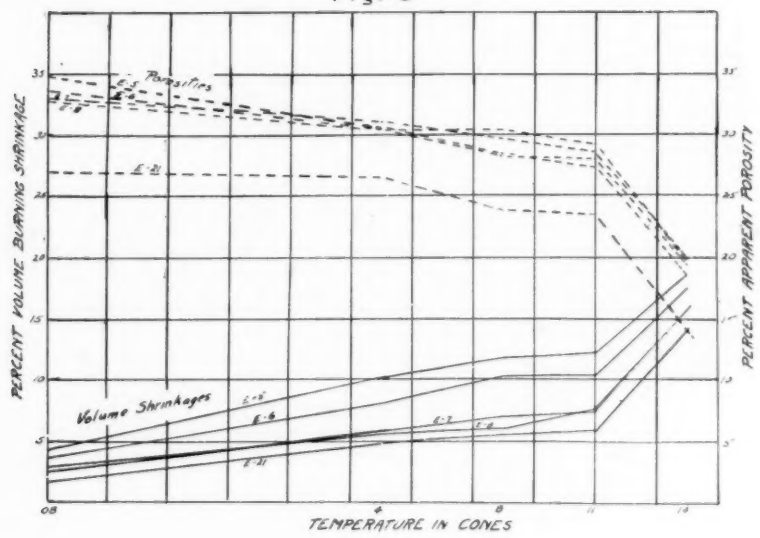

PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
Porosities
Volume Shrinkages
TEMPERATURE IN CONES

Fig. 9

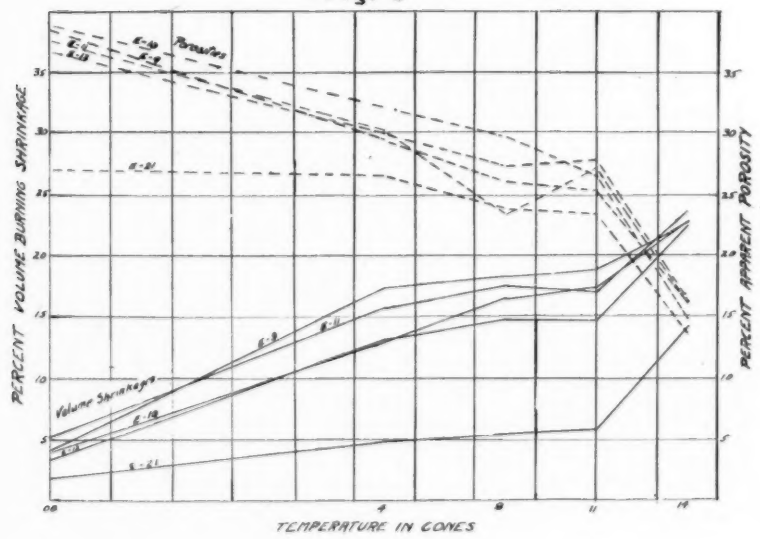

PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
Porosities
Volume Shrinkages
TEMPERATURE IN CONES

Fig. 10

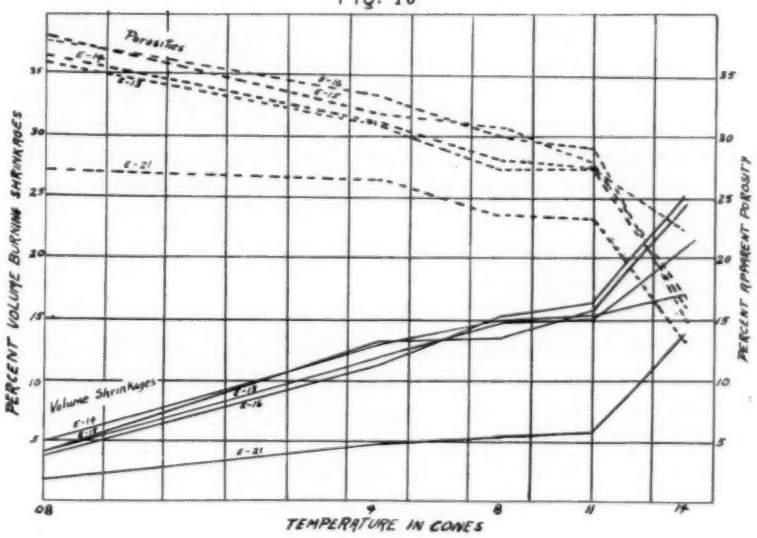
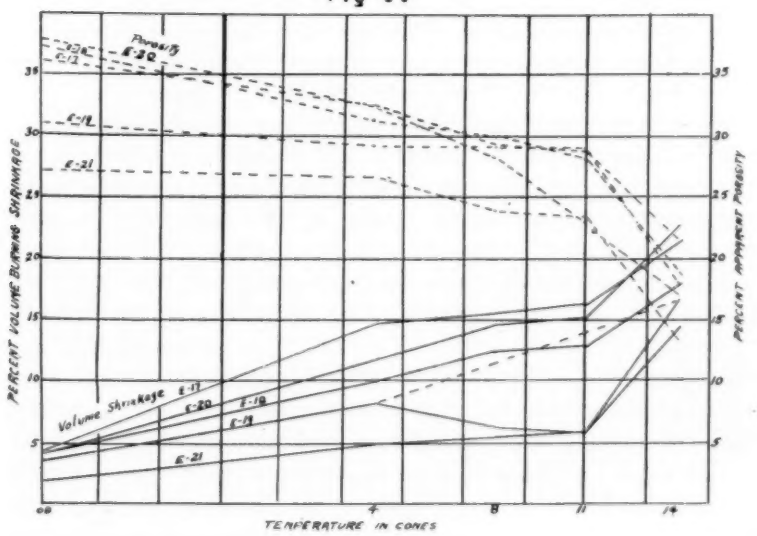

Porosities
E-18
E-17
E-16
E-21
Volume Shrinkages
E-17
E-18
E-16
E-21
PERCENT VOLUME BURNING SHRINKAGES
PERCENT APPARENT POROSITY
TEMPERATURE IN CONES
08
4
8
11
14
35
30
25
20
15
10
5

Fig 11


Porosity
E-20
E-19
E-21
Volume Shrinkage
E-17
E-20
E-18
E-19
E-21
PERCENT VOLUME BURNING SHRINKAGE
PERCENT APPARENT POROSITY
TEMPERATURE IN CONES
08
4
8
11
14
35
30
25
20
15
10
5

E-6.—Same as E-5, but somewhat more porous. White layer between glass and clay, somewhat more pronounced though attack has been slight.

E-7.—Very similar to G. A. body, both as regards glass attack and density.

E-8.—Again similar to G. A. body but showing somewhat less attack by glass than corresponding composition with Tennessee clay E-4.

E-9.—Somewhat heavier glass attack than G. A. body, but of much lighter color.

E-10.—Much more glass attack than G. A. body.

E-11.—Glass attack still more pronounced and white layer between glass and clay, more than in G. A. body.

E-12.—Glass attack very pronounced.

E-13.—Glass attack heavier than in G. A. body.

E-14.—Glass attack heavier than in G. A. body but not as much as in E-13.

E-15.—Glass attack heavier than in G. A. body; structure rough.

E-16.—Glass attack quite heavy and glass colored dark green by iron.

E-17.—Glass attack heavier than with G. A. body, but color of glass much lighter; structure quite porous.

E-18.—Glass attack slightly more than in G. A. body, but color much lighter. Body structure fairly dense.

E-19.—Glass attack slightly heavier than in G. A. body.

E-20.—Glass attack quite severe and glass a very dark color.

It appears from the corrosion test that the difference between the American clay bodies and the G. A. clay is not very marked except in the case of the New Jersey saggar clay. It would appear from this that the primary advantage of the G. A. clay lies not so much in its resistance to corrosion, as in its low volume changes upon firing and its rigidity at furnace temperatures and under load condition. It would appear, therefore, that from the standpoint of resistance to corrosion, our American clays are at least equal or superior to the G. A. clay. The conclusion must be reached therefore, that we can not only expect to reproduce a type of clay equivalent to the G. A. clay, but that we can obtain bodies superior to it. The latter condition can

be reached with still greater certainty by increasing the previous heat treatment of the pots or tank blocks, that is, by raising the arch temperatures and striving for greater uniformity in temperature during this preliminary burning. It is expected that a more comprehensive study of the resistance to corrosion will be made, employing glass batches attacking the structure more vigorously and discriminating more sharply between the different materials.

Discussion

GEO. A. LOOMIS: This is a very interesting and valuable paper and the data presented represent a great deal of work.

In regard to the corrosion test, I believe that twelve hours is much too short a time for such a test to differentiate sufficiently between the different mixtures. I believe it is imperative to run such a test at least twenty-four hours at the final temperature to get a proper differentiation. I realize that in isolated laboratories it is not always possible to have compressed air continuously for the burners to maintain the high temperature for this length of time. That is no doubt the reason that the time was not extended in this case. Extending the time for the test is, however, much to be preferred to using a more corrosive glass than that used.

C. W. BERRY: Mr. Fuller's paper should prove of value to the Glass manufacturer and also the pot manufacturer, in that it shows the existence of satisfactory American clays. Under the present methods of arching pots, the silicious mixture generally gives the best service, although with proper temperatures in the arches, the aluminous type of pot is satisfactory.

During the past year the writer has witnessed a number of tests made on an aluminous type of pot. This mixture showed great resistance to corrosion, but owing to the low temperatures employed in the arches considerable difficulty was experienced after pots were placed in the furnaces.

At one plant the pots were heated to 2700° F. in the arches and the pots gave excellent service. This shows the need of improved arches and higher temperatures.

POROSITY: I. PURPOSE OF THE INVESTIGATION II. POROSITY AND THE MECHANISM OF ABSORPTION[1]

By Edward W. Washburn

ABSTRACT

Significance of porosity.—This is briefly outlined.

Standard reference method.—The requirements of an *engineering* testing method are simplicity, rapidity and convenience with no more accuracy than practical considerations justify. There should, however, be also available a *standard reference method*, that is, a laboratory method where accuracy is the main requirement.

Types of pores.—Six types of pores are described and the *process of absorption* under various conditions is analyzed.

Definitions.—Definitions are proposed for the terms, *permeability, penetrability, absorptivity, porosity, bulk density, closed-pore space* and *"cracks"* and *"holes,"* and methods of measurement are suggested.

Introduction

1. Purpose of the Investigation.—The critical examination of the methods of testing which are employed for securing those data which characterize and measure the qualities or behavior of the raw materials or the products of any branch of technology is justly regarded as one of the important fields of research of every applied science. In studying and interpreting the behavior of ceramic products during drying and firing, the determination of the porosity and of the changes which it undergoes, especially during the firing operation, is an important factor. Porosity also has an important bearing upon the strength of the body, upon its behavior as an adsorbent, and upon its resistance to weathering, shock, abrasion, erosion, slagging, temperature strains, discoloring agents, efflorescence, chemical attack by gases and liquids, and the destructive action of fungus growths,[2] as well as upon the values of certain physical proper-

[1] Received May 1, 1921.

[2] Scott, James, "Tile Fungus," *Brit. Clayworker*, **18,** 138 (1920).

ties of the body such as its bulk density, its strength as a dielectric, its permeability, and its thermal and electrical conductivities.

An examination of the literature dealing with this subject reveals the fact that no adequate systematic study seems ever to have been made of the nature and magnitude of the errors involved in determining porosity by the commonly used methods of absorption. The present series of investigations was undertaken for the purpose of throwing some light upon these questions and of evolving, if possible, a procedure for porosity measurements which should meet the requirements of a *standard reference method.*

In order that the purpose of the investigation may be clearly understood a brief explanation of term *standard reference method* and of its relation to what may be termed practical or engineering standard methods is desirable. The important characteristics of an *engineering* testing method are simplicity, rapidity, and convenience. The degree of accuracy attainable in the method need only be that demanded or justified by the nature of the materials to be tested and the purpose of the information sought. A higher degree of accuracy than this is undesirable, if its attainment involves an essential sacrifice of rapidity or convenience. Thus the measurement of porosity by the comparatively rough and inexact method of soaking in water might give all the information necessary (or all the information which the variability of the material under examination would justify) in the case of common brick for the purposes of the structural engineer, although the same method would obviously be quite useless if applied, for example, to a ball mill body or to a piece of electrical porcelain.

In order to ascertain the accuracy of a proposed engineering method and thus to form an opinion of its availability for a given class of materials, it is desirable, in many instances, to have available also what we have termed a *standard reference method,* that is, a laboratory method in which *accuracy* is the principal requirement and rapidity and convenience, while important, are only secondary. Such a method should preferably be applicable to as great a variety of materials as possible. With this

purpose in mind we shall, in the subsequent papers of this series, proceed to examine the various methods which have been proposed and used for determining porosity, in order to ascertain the nature and magnitudes of the errors which they involve and, in the light of the information thus obtained, to devise an appropriate standard reference method. The remainder of the present paper will be devoted to an analysis of the mechanism of absorption and to proposed definitions of terms.

II. Porosity and the Mechanism of Absorption

2. The Factors Involved.—Before proceeding to an examination of the methods of measuring porosity and to the consideration of the problems connected with the formulation of a "standard reference method" it is desirable to have a clear concept of the exact nature of the quantity which such a method is intended to measure and of its relation to the associated properties of closed-pore space, permeability, penetrability, absorptivity on immersion, bulk density, true density, cracks, and holes.

This subject may perhaps be best approached by attempting to form a mental picture of the various types of pores which may exist in a body.

3. Types of Pores.—In figure 1 are depicted a variety of types of pores which one can imagine as possible forms produced during the firing of a ceramic body. For convenience in reference, the types shown may be designated as follows:

(1) THE CLOSED OR BUBBLE PORE, *a*.

(2) THE CHANNEL PORE.—These are of two principal types, the vertical, b_1, and the horizontal, b_2, with various intermediate inclined forms. Belonging to this type are also the connecting pores which exist between the particles of a mass made up of grains in simple contact with one another, a simple example of which is a heap of sand grains. The channel pores connect with one another so as to form a passage way through the body.

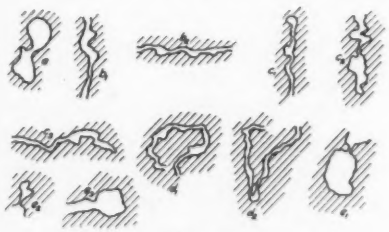


FIG. 1.—Types of pores.

(3) THE BLIND-ALLEY PORE.—These are of three principal types, the upright, c_1, the inverted, c_2, and the horizontal, c_3, with the various intermediate inclined forms.

(4) THE LOOP PORE.—These may be either upright, d_1, inverted, d_2, horizontal, or of an intermediate inclined type.

(5) THE POCKET PORE.—These are fairly large pores with short necks. The necks may be wide as in e_3, narrow as in e_1, and e_2, or so narrow as to cause the pocket to approach the closed-pore type in behavior.

(6) THE MICROPORE.—This type of pore, which is not represented in the figure, includes all pores with diameters so small that they can not be filled by a liquid in any reasonable time of soaking. The diameters of many of them approach molecular dimensions and the type itself gradually merges into the intermolecular pore space. There is probably in many cases also no sharp division between this type and the larger pores whose volume is included in the measured pore volume of the material.

A great variety of combinations and modifications of the above types are obviously possible, and even with regard to the pictured forms shown in Fig. 1, it must be remembered that they actually exist in three dimensions and not in two as represented. The types shown will, however, suffice to make clear the various possible types of behavior.

4. Permeability.—It seems desirable to restrict the use of this term to represent the readiness with which the body permits a fluid to flow through it from one side to the other. The permeability may be measured by the rate at which some standard fluid, either a gas or a liquid, flows through unit area and unit thickness of the body under some specified pressure head. From this definition, it is evident that only the channel pores are active in determining the permeability of a body, with the possible exception of some of the loop pores which might happen to be so located with reference to the direction of flow as to function to a slight extent as channel pores. The permeability will, therefore, be determined by the number and dimensions of the channel pores and by their locations with respect to the line of flow.[3]

[3] For examples of the measurement of the permeability of refractory materials see Wologdine, S., *Electrochem. Met. Ind.*, **7**, 1433 (1909).

5. Penetrability.—In connection with determining the resistance of a body to weathering and to the action of slagging and discoloring agents, it has been proposed to measure a property which may be appropriately designated as the penetrability of the body. As a *physical* quantity, it may be qualitatively defined as the ease with which a liquid is drawn into the pores of the body by capillary action, without attendant chemical action between the body and the liquid.

A quantitative definition is possible only when agreement is reached as to the method to be used in measuring the property in question. It might, for example, be measured in the following way: By the amount of a standard liquid which, in a stated time, a standard test piece will absorb, at a stated temperature, when a stated surface of the piece, in a determined position (horizontal or vertical, and, if horizontal, then a lower not an upper surface), is kept in contact with the liquid, and the air surrounding the test piece is kept saturated.

The only class of pores which could be completely filled by capillary forces alone would be the channel pores. All of the other classes except the closed pores would, however, be *partially* filled. Not all of the channel pores would necessarily be completely filled, however, since the height of the capillary rise in a vertical channel pore is limited by its diameter.

6. Absorptivity on Complete Immersion.—The amount of liquid absorbed by a test piece, completely immersed with all of its surface pores open, would be determined by a combination of its penetrability, its permeability, and its porosity. The liquid would flow upward through, and eventually completely fill, the channel pores because of the pressure difference on the upper and lower faces of the piece. It would be drawn in laterally by the capillary forces, and these forces would eventually *partially* fill the remaining types of pores except the closed pores. The air entrapped in the partially filled pores would then dissolve slowly in the liquid and diffuse still more slowly outward and eventually escape, thus permitting these pores after a long period of time to become completely filled with the liquid. The method of simple immersion (*i. e.*, without boiling)

whether *in vacuo* or not, does not,[4] therefore, measure any accurately definable physical property of the material, unless the immersion is continued for the *very* long period necessary to fill the pores by the slow process of solution and diffusion of the air, in which case the method measures the porosity.[5] The removal of the air from a pocket pore with a narrow opening would be especially slow by this process. The absorptivity on immersion will thus be a function of the time of immersion and of the surface tension and viscosity of the immersion liquid.

7. Porosity.—By the term porosity we shall understand the open-pore porosity. It differs from the preceding properties in that it includes the total volume of all types of pores except part of the micro-pores and except those closed pores having walls of sufficient strength to withstand the treatment to which the test piece is subjected during the porosity measurement. Any method employed for measuring it must be one which will insure the complete filling of all these pores by the fluid employed.

8. Bulk Density and True Density.—The term "bulk density" (called also "apparent density") may be conveniently employed to designate the ratio of the mass of the test piece to its volume. It is conveniently and accurately determined by weighing the completely saturated test piece suspended in any suitable liquid or by means of a volumeter method using mercury as a liquid.

The true density is most conveniently measured by the pycnometer method applied to the finely powdered material. With suitable precautions, which have already been carefully worked

[4] Unless the vacuum is high enough to remove substantially all the air. The use of a moderate vacuum would have no practical value, since it would not hasten the slow stage of the absorption process.

[5] In spite of the fact that the result obtained varies with the time of immersion it is nevertheless conceivable that by specifying a standard immersion procedure, the measurement of absorptivity under these conditions might give an excellent indication of the probable resistance of the body to freezing and thawing of absorbed water, for example. In other words a testing method might give a result which would indicate the general behavior of a material under certain conditions, even though it did not measure any quantitatively definable physical property, in the strict sense of the term.

out,[6] the true density can, if desired, be determined with a precision of better than 0.1 per cent, provided closed pores smaller than the particles of the powder are not present.

9. Closed-Pore Space.—This is calculated from the true density, the bulk density, and the porosity. When so calculated it includes only those closed pores whose walls are strong enough to withstand the temperature and pressure differences which are produced by the procedure employed in measuring the porosity. In practice it would also, of course, include part of any open pores which did not become completely filled with the absorption liquid. This method of determining closed-pore space is, of course, reliable only if the fineness of the grinding employed in connection with the true-density measurement is sufficient to rupture all the closed pores.

10. Cracks and Holes.—Cracks and holes are spaces in a body so large that they should not be counted in, in computing its porosity and the method used for measuring porosity should, if possible, be one which can be depended upon to reject such spaces. Now the maximum sized space which would be classed as a pore obviously varies with the nature of the ware. Thus most of the actual pores of a "non-pareil" insulating brick are so large that if they were found in a piece of porcelain they would be classed as holes.

It is obvious, therefore, that a standard reference method for measuring porosity should be of such a character that the maximum size of opening to be included as part of the pore space should be under the control of the operator. Methods of accomplishing this will be discussed later.

(To be continued)

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[6] See, for example, Johnston, J., and Adams, L. H., *Jour. Amer. Chem. Soc.*, **34,** 572 (1912); cf. Cude and Hullett, *Ibid.*, **42,** 400 (1920).

DISCOLORATION OF WHITEWARE FIRED IN CARBORUNDUM SAGGARS[1]

By H. Spurrier

ABSTRACT

The author advances the hypothesis that iron carbonyl, formed by reaction between CO and iron present in the carborundum saggars, condenses in the central portion of the bung, trickles down over the ware and is later decomposed, giving iron and free carbon, which are absorbed and color the ware. Laboratory experiments completely confirmed this hypothesis.

Many of those who have used carborundum saggars for whiteware have experienced annoyance from discoloration of the ware. The manner in which this discoloration takes place presents some peculiarities which on careful examination furnish the clue to the cause of the trouble.

It is not uncommon to find a saggar of spark plug blanks uncolored or only slightly colored on the outer rings, but with a central portion exhibiting a rather beautiful, but very undesirable range of color from dove gray to dark battleship gray, or even nearly black, on the upper portion; while the lower portion shows a rust red and no mottle. The uppermost and lowermost saggars of a bung never show these characteristics to nearly the extent exhibited by the saggars in between.

On inspecting some of the discolored ware it will be noticed that the plugs in many cases appear to have some ferruginous liquid spattered upon them, which has trickled down the nearly straight sides to a projecting ring or a recess, there to gradually flow around the ring till a sufficient accumulation forces it to find a channel by which to flow still further down to the bottom of the plug where it again accumulates. Firing will subsequently oxidize the ferruginous liquid and leave a path, rust red in color, and fluxed permanently into the glaze. The bore hole of these plugs is also colored but of a reddish fawn evidently the product of the gray and the incompletely oxidized iron.

[1] Received March 11, 1921

These appearances are so suggestive as to unavoidably lead to the hypothesis that the carborundum saggars are responsible for the discoloration and one naturally begins to speculate as to some rationale that will fit the observed phenomena to the speculation.

It is well-known that carbon monoxide at about 70–80 °C will, in the presence of finely divided iron, produce volatile carbonyls of which two are well-known: Tetracarbonyl, $Fe(CO)_4$, and pentacarbonyl, $Fe(CO)_5$; that at higher temperatures these carbonyls are decomposed into iron and carbon monoxide,[1] the finely divided iron quickly oxidizing if circumstances permit.[2]

Supposing this to be taking place, how does it fit the evidence? Assuming the kiln placed and firing commenced, the temperature rises to the necessary 80 °C, the top and bottom saggars of the bungs receive heat from the sides and from the top and bottom respectively also, and the intermediate bungs are heated mainly from the sides, the centers remaining relatively cool for a period.

We have therefore the condition that explains the ring of nearly uncolored plugs in the intermediate saggars, which does not appear or is much less pronounced in the top and bottom, saggars. The intermediate saggars rise to or above 80 °C on the outside, the centres remaining cooler. The formation of iron carbonyl (assuming of course the presence of the necessary metallic iron and carbon monoxide in the system) commences, and it is driven toward the cooler portion which is at the centre. Here the carbonyl vapors, probably assisted by whatever plus pressure may exist,[3] tend to condense, and the liquid gradually accumulating trickles down the plugs producing the lines of flow previously described. As the temperature at the center rises to the decomposition point of the iron carbonyl, it must be decomposed here, as there is no cooler point for its condensation. The iron is left as a residue and some of the carbon is

[1] This is also true of nickel and forms the basis of Ludwig Mond's process for the production of metallic nickel.

[2] The red discoloration found on steatite gas tips is caused by the breakdown of iron carbonyl (formed from the carbon monoxide) upon reaching the heat of the gas tip.

[3] The formation of iron carbonyls is much assisted by pressure which moreover stabilizes them somewhat against decomposition by heat.

absorbed, producing the graduation of the gray shades. As the outer rings of plugs in the saggars are heated at the start the above-mentioned reaction is here largely avoided. The iron carbonyl does not condense on them and the ware consequently remains white or much lighter in color.

So much for the thermo-chemical behavior; but does the iron exist in the carborundum saggar? And if so, would carbonyl be formed under the conditions? And if formed would it have the effect noted?

The presence of finely divided iron in the carborundum saggars may be proved by crushing portions of new saggars in a Wedgwood mortar and subjecting them to the action of an electromagnet, the poles of which are protected with a thin paper sheath. On interruption of the current the metal particles are dropped at once. Magnetic separation is carried on till the metal particles are quite clean and free from foreign matter. The identity of the magnetic concentrate thus cleaned has been established by chemical tests (nearly complete solubility in hydrochloric acid and so forth) and by microscope observation.

The existence of carbon monoxide needs no demonstration because its presence is unavoidable. Consequently the necessary conditions exist.

It now remains to prove that the saggar material heated gently in the presence of carbon monoxide will actually yield iron carbonyl and that iron carbonyl will produce the effect noted. The procedure is as follows. Partly fill a quartz conbustion tube with the saggar material crushed as before, then insert a plug of glass wool and charge a portion of the tube with granules of charcoal. The portion charged with carborundum can be kept at the desired temperature by packing filter paper around it and allowing warm water to continually flow over it. Heat the portion containing the charcoal to full redness and pass through a gentle stream of dried and purified carbon dioxide. The familiar reaction of the reduction of carbon dioxide to carbon monoxide takes place.

The exit end of the quartz tube should be furnished with a small condenser kept below 70°C with a cooling mixture if the hydrant water is too warm to use. After a considerable time

the above experimental procedure yielded a very small amount of liquid which, on heating in a tube under a slight pressure, decomposed, leaving a small residue. The issuing gas reacted with palladium paper indicating carbon monoxide. On breaking the tube the residue was found by chemical and microscopic tests to be ferruginous.

The next step was to actually produce the discolored body by the above means, and, owing to the minute quantity of iron carbonyl produced in the previous test, it was necessary to use a somewhat different method. Accordingly the tube was set up as before, but without the small condenser, a stopper and educt tube taking its place. A small piece of glazed and unfired ware was placed between the carborundum and the stopper, a space of about 12 inches. The portion of the tube holding the charcoal was well heated, no carbon dioxide being admitted. The tube was then evacuated, the portion of the tube charged with carborundum being kept between 70–80°C. The carbon dioxide was then very slowly admitted till the carbon dioxide generator showed a slight pressure, evacuation of course being stopped. The portion holding the clay ware was next gradually heated, heating being continued with a large blast lamp urged to the limit. After two hours the heating was discontinued and the apparatus allowed to cool. When the test piece was taken out, although underburned, it showed the characteristic discoloration, the gray color was pronounced, the mottling of gray and ruddy color, slight, but in evidence. Similar pieces fired in the same tube without the carborundum showed no discoloration and the results compel the only possible conclusion, that the discoloration is due to the formation of iron carbonyl, which is made possible by the free iron in the crushed carborundum.

Carborundum carefully boiled for a long time with hydrochloric acid washed and dried does not, on treating as above, result in discolored ware in the quartz tube.

Although the writer has not personally proved the existence of free carbon in carborundum, he understands that it may sometimes be present and in such cases the formation of iron carbonyl would be much facilitated.

DETROIT, MICH

NOTE ON THE DISSOCIATION OF FERRIC OXIDE DISSOLVED IN GLASS AND ITS RELATION TO THE COLOR OF IRON-BEARING GLASSES.[1]

By J. C. Hostetter and H. S. Roberts

ABSTRACT

It is shown that above 1300°C, Fe_2O_3 is dissociated appreciably with the evolution of O_2 and the formation of the corresponding quantities of FeO. The same reaction is found to take place in glasses. The following percentages of Fe as FeO based on the total Fe present illustrate the extent of this dissociation in glasses. Diopside glass ($CaSiO_3.MgSiO_3$) 1400° C, 20%; 1500° C, 30%; 1600° C, 45%. 0.6 K_2O—0.4 CaO—$2SiO_2$, 1310° C, 5.8%; 1555° C, 10.1%. 0.3 K_2O—0.3Na_2O—0.4 CaO—$2SiO_2$, 1020° C, 4.1%. In the two latter glasses a dissociation of 10% produced a bright green color: the glass with a dissociation of 4.1% was yellow. Iron compounds in a glass may therefore be "reduced" by heating to a high temperature under oxidizing conditions. The intensity of the color due to iron is closely related to the composition of the glass, but data on this point are not sufficiently complete to warrant generalizations.

General Effects of Iron in Glass

Iron, in some form, is present as an impurity in every raw material used in the manufacture of optical glass. When the quality of raw materials and pots is otherwise satisfactory, the color developed by iron in glass is the most important factor in lowering transmission. The actual iron content of precision optical glass varies from 0.01 per cent to 0.05 per cent (expressed as Fe_2O_3) even when made from the purest raw materials obtainable and melted in resistant pots of low iron content. The absorption of light caused by even these small amounts of iron is significant and easily measurable, but the relation between total transmission and iron content is obscured somewhat because of the two co-existing states of oxidation in which iron is

[1] Received September 1, 1921. Read at the Joint Meeting of the Glass Division of the American Ceramic Society and the English Society of Glass Technology in Pittsburgh, September 2, 1920.

found in the glass and the difference in absorption produced by each state, ferric iron generally giving a yellowish tint and ferrous iron producing green. The effect on transmission of the same approximate total iron content varies in the different types of glass; the transmission of borosilicates being in general higher than that of flints, ordinary crowns or barium crowns. The statement that soda increases color when substituted for potash in glass is frequently made, although detailed determinations of iron contents of materials and of final glasses are usually lacking when this statement appears.

In order to obtain specific information concerning the effect of iron when present in the batch in different forms, an extensive series of melts on experimental glasses was made[1] by our colleagues, F. E. Wright and N. L. Bowen, who introduced iron into glass batches as metallic iron, magnetite, ferrous carbonate, and ferric oxide. The ferrous and ferric iron in these glasses was subsequently determined[2] and transmissions measured. The relation between ferrous and ferric iron in any particular series of glasses did not appear in general to be dependent upon the form in which the iron was introduced into the batch, and this condition indicated immediately that some equilibrium was approximately established between these two states of oxidation. The melts were made in small open pots (12 × 12 inches) in a recuperative furnace which was fired with artificial gas, and the first conclusion regarding the state of oxidation of the iron in the glass was naturally that the ratio between ferrous and ferric iron was that developed by the action of the products of combustion.

In the present paper there is expressed another point of view which is not intended to supplant the idea that the ratio of ferrous to ferric iron in a glass is dependent upon the composition of the products of combustion above the melt, but rather to supplement this conclusion by showing that ferric oxide dissolved in glass can be "reduced" to the ferrous state when heated in pure air, that is, under "oxidizing" conditions.

[1] At the Bausch and Lomb Optical Company.

[2] Some of these analyses have been published by Ferguson and Hostetter. THIS JOURNAL, **2**, 608–621 (1919).

Dissociation of Ferric Oxide

When ferric oxide is heated *in vacuo* it gives off oxygen. The relations between oxygen pressure, temperature and composition have been investigated.[1] As oxygen is evolved an equivalent amount of "ferrous oxide" is formed which in all probability dissolves in the ferric oxide to form a solid solution. This conclusion is based upon microscopical evidence and also on the characteristics of the isotherms for this system. Figure 1 shows the characteristic shape of the isotherms actually secured and also, in the dotted lines, the probable shape of the isotherms at other temperatures. It has also been determined that ferric oxide, even when heated in air, loses some of its oxygen and forms a corresponding amount of ferrous oxide. A slight oxygen loss takes place as low as 1000°C and this loss increases at higher temperatures until melting takes place at 1590°C, when the product consists of magnetite which contains 31.1 per cent FeO. Oxygen lost at high temperatures is reabsorbed at lower temperatures if the charge is cooled slowly—thus indicating that the action is reversible.

The formation of "ferrous oxide" under these conditions—by dissociation—is to be carefully distinguished from the reduction of ferric oxide by such constituents of furnace gases as carbon monoxide and hydrocarbons. These gases not only dilute the oxygen, thereby lowering its partial pressure, but they remove oxygen from the already reduced ferric oxide and combine with it chemically, thus increasing the amount of "ferrous oxide." The formation of ferrous oxide by dissociation in the first case takes place under what are ordinarily considered to be oxidizing conditions. In fact, it has been shown that ferric oxide loses weight with attendant formation of ferrous oxide in one atmosphere of oxygen at 1300°.[2]

The order of magnitude of the amount of ferrous oxide formed when ferric oxide is heated to different temperatures in air is shown in table 1. The data presented here as series 1 have been recalculated from those given in the paper last quoted. The data of series 2 represent ignition experiments under differ-

[1] Sosman and Hostetter, *J. Am. Chem. Soc.*, **38**, 807–833 (1916).

[2] Hostetter and Sosman, *Ibid.*, **38**, 1188–1198 (1916).

TABLE I.—SYSTEM Fe_2O_3–Fe_3O_4

Ferrous oxide in equilibrium at different temperatures with the oxygen pressure in air (152 mm.).

The first three values under "Per cent FeO" resulted from series 1, the other six from series 2.

Temperature °C	1150	1200	1250	1283	1350	1377	1401	1450	1590
Per cent FeO	0.053	0.067	0.082	0.4	0.8	1.85	14.1	19.9	31.1

ent conditions. The data of series 1 were obtained by following changes in weight that occurred when ferric oxide was heated alternately in air and oxygen at certain temperatures under controlled conditions that ensured freedom from reducing material of any description. The charges were held in "alundum" boats placed in a porcelain tube heated electrically and the air and oxygen used were specially purified. In series 2 the charges were held in a platinum crucible heated in an electric furnace for 1½ hours at the given temperature, while a slow stream of air was passed over the charge. The thermocouple in this series was inserted in the charge. Since platinum reduces the oxide of iron in contact with it at high temperatures,[1] suitable precautions were taken to ensure the removal of the layers in contact with the crucible before analyses were made to determine the amount of ferrous oxide formed; weight changes were not followed here.

The data of series 1 and 2 are plotted in figure 2. Such a curve represents the isobar for the system Fe_2O_3–Fe_3O_4–O_2 at 152 mm. oxygen pressure which is marked by the heavy horizontal line in figure 1. The general shape of the curve of figure 2 is in satisfactory agreement with that indicated by the points on the 152 mm. isobar if the isotherms for higher temperatures follow the same curvature as those for 1100° and 1200°.

Dissociation of Ferric Oxide in Clays

Certain qualitative results obtained by Kinnison[2] may be mentioned here. As far as we know, they are the only results recorded which have been obtained under oxidizing conditions. He subjected four different clay mixtures containing 5 per cent

[1] Sosman and Hostetter, *J. Wash. Acad. Sci.*, **5**, 293–303 (1915).

[2] *Trans. Am. Ceram. Soc.*, **16**, 136 (1914).

Fe_2O_3 to high temperatures under oxidizing conditions (air) and noted the colors of the slabs after exposure to a definite temperature for 1.5 hours. At 1100° all of the ignited specimens were red. At 1250° one of the tests yielded a greenish brown glass, while the other three varied from cream color to pink.

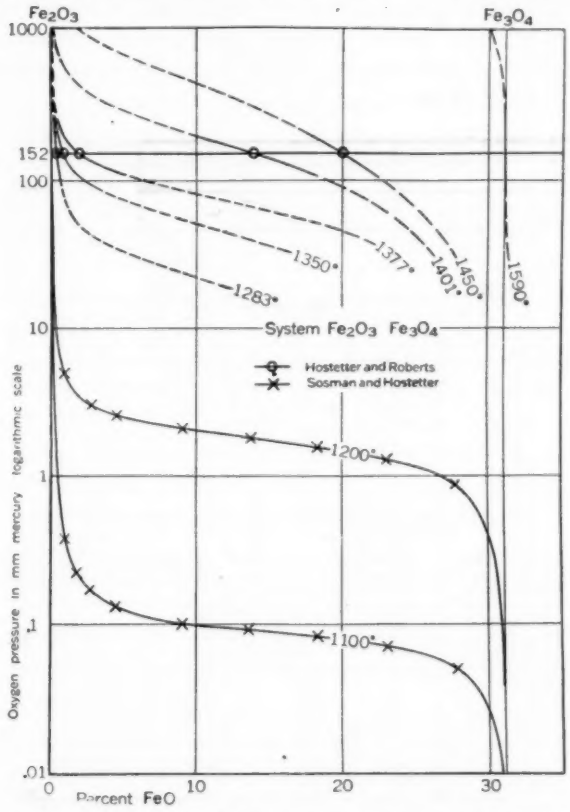

FIG. 1.—*Iron oxides alone.* Dissociation pressure and composition (isotherms).

At 1400°, the maximum temperature, another mixture yielded a black glass and the other two were black in color but not vitrified. As the temperature was increased from 1100° it was evident that reduction of the ferric iron causing the initial red color had taken place and that the ferrous iron so formed was responsi-

ble for the darkening in color. Kinnison attributed the formation of ferrous oxide to a "transition" of the higher to the lower oxide, but, as shown above, ferric oxide dissociates over a considerable range in temperature with the formation of a solid solution instead of yielding a second phase. The black specimens obtained by Kinnison were clay bodies mixed with solid solutions of ferrous oxide in ferric oxide with possibly local zones of fusion around each particle of iron oxide. He points out that ferrous iron yields a more fluid glass than ferric iron.

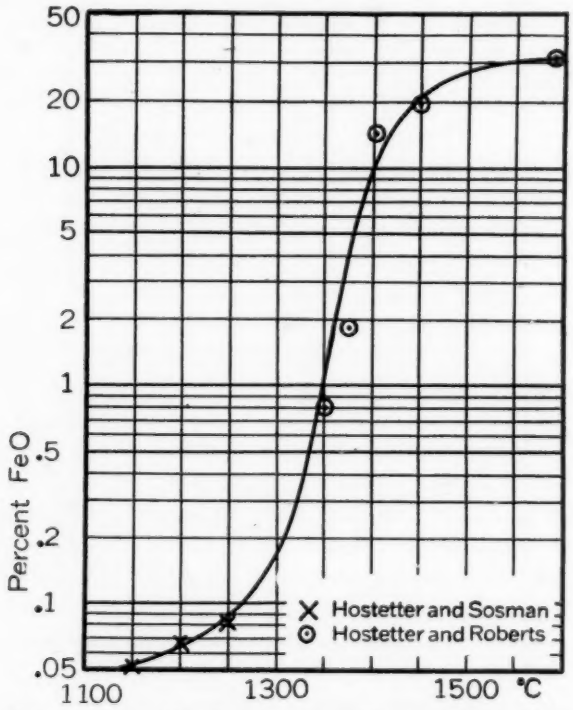

FIG. 2.—*Iron oxides alone.* Composition and temperature for charges heated in air (152 mm. isobar).

Dissociation of Ferric Oxide Dissolved in Glass

It can also be readily enough shown that ferric oxide dissolved in glass loses oxygen at higher temperatures, even under

the oxygen pressure of the air. This is evidenced not only by the change in color of the glass from yellow to green, as the glass is fined at successively higher temperatures, but also by actual chemical analysis of the glass. In the experimental work which follows it will be shown that ferric oxide dissolved in a glass at a comparatively low temperature loses oxygen when the glass is subsequently heated to a higher temperature in pure air. Some of the experiments that will be described below were carried out in collaboration with R. B. Sosman about 1913 in preliminary work on the system diopside: ferrous silicate. The other experiments were made more recently in order to demonstrate the applicability of these results to actual glass-making processes.

The actual dissociation in any particular case will depend on the composition of the glass in which the ferric oxide is dissolved. Any variation in composition of the glass will be reflected in the dissociation pressure at a given temperature since a change either in the number or character of the components, or the concentration in a given system, will necessarily raise or lower the pressure in a manner depending on the characteristics of the system.

The method of experimentation was, briefly, to make up a glass containing ferric oxide at a low temperature. Subsequently a portion of this glass was heated to a definite higher temperature in a stream of air for a fixed length of time. The crucible containing the glass was then removed from the furnace and the melt quenched in water. The color of the glass was noted and then the total iron and ferrous iron contents were determined.

In the first series, the glass used consisted of diopside ($CaSiO_3$.-$MgSiO_3$) to which was added 8 per cent of iron as ferric oxide, and the equivalent amount of silica to make ferrous silicate, $FeSiO_3$. The results obtained with this glass are presented in table 2. We may note here that the glass fined at about 1400° contains approximately 20 per cent of the total iron in the ferrous condition; the color of this glass is decidedly brown. At 1589°, however, no less than 41 per cent of the total iron is ferrous and the color is green. Between these two extremes the color varies from brown through olive green to clear green. The relation between percentage of ferrous oxide and temperature is shown in figure 3.

TABLE 2

Change in state of oxidation of iron oxide dissolved in diopside ($CaSiO_3$-$MgSiO_3$) glass on heating to different temperatures in air.

Initial total iron content of glass, 8.08 per cent.

Minutes at maximum temperature	Maximum temperature, °C	Ferrous iron % of total iron	Color of glass
—	1397	20.4	brown
16	1407	21.6	——
—	1422	18.5	——
70	1431	26.0	olive
20	1526	25.9	——
—	1526	39.4	——
20	1530	29.3	——
—	1530	30.7	——
15	1589	41.2	bright green

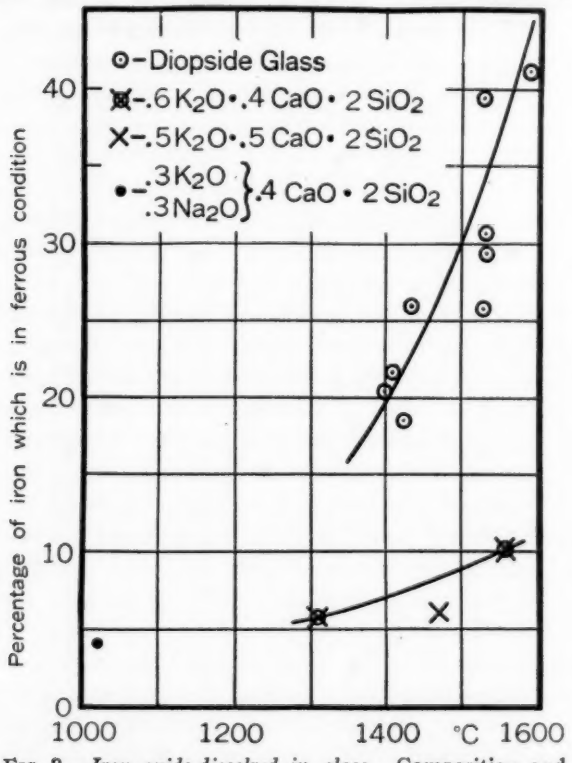

FIG. 3.—*Iron oxide dissolved in glass.* Composition and temperature for charges heated in air (152 mm. isobar).

A series of crown glasses was made up and similar tests made at different temperatures. The compositions and results obtained are given in table 3. It will be noticed here that the percentage of ferrous iron formed at a given temperature is

TABLE 3

Ferrous iron in glasses fined at various temperatures.
Initial total iron content of glass, 0.7 per cent.

No. of glass	Composition		Time at high temperature hrs.	Maximum temperature °C	Ferrous iron % of total iron	Comparative color of glass
R478	$0.3K_2O$ $.3Na_2O$ $.4CaO$	$2SiO_2$	48	1020	4.1	lemon yellow
6377A	$0.6K_2O$ $.4CaO$	$2SiO_2$	5	1310	5.8	greenish yellow
6368A	$.5K_2O$ $.5CaO$	$2SiO_2$	1	1470	6.0	yellowish green
6372A	$.6K_2O$ $0.4CaO$	$2SiO_2$	½	1555	10.1	bright green

of an entirely different magnitude from that obtained with the diopside glasses. The amount of oxygen lost at a given temperature depends upon the composition of the glass and will change with each variation in composition. It may be noted here that when these glasses were fined at 950° to 1000°, the color was lemon yellow. After being held for 13 minutes at 1555° the color was bright green, even though only 10 per cent of the total iron was in the ferrous condition.

Discussion of Experimental Results

From the results presented it is apparent that the formation of ferrous oxide in a glass may result from dissociation of the higher oxide as well as from possible reduction by gases. The two causes may operate in a given case, one or the other predominating, depending on conditions. However, since, in the case of dissociation, oxygen is reabsorbed as the melt cools, it is obvious that, in the later stages of stirring optical glass when the fire is off, there are obtained excellent conditions for the attainment of equilibrium with the oxygen of the air. Cooling a glass melt in air without stirring presents less opportunity

for the absorption of oxygen, but the case is quite different with the usual practice for optical glass. It is appropriate to note here that the amount of ferrous iron found in the experimental optical glass melts mentioned earlier in this paper ranged from 7 to 38 per cent of the total iron—the same range of values covered by the experiments presented in this paper.

It is well known that certain types of optical glass generally possess a higher transmission than other types. Aside from the facts that the denser glasses give a more intense color with a given impurity than glasses of less density (and lower index of refraction) and that the different alkalies give different colors with the same impurity, it is also common experience that borosilicates, for instance, usually possess higher transmission than barium crowns. A pot of the latter glass at the Bausch and Lomb Optical Company, pronounced "poor color" when examined by Mr. V. Martin, the experienced glass-maker in charge, was later found to contain iron equivalent to 0.075 per cent Fe_2O_3. However, a pot of borosilicate, of "good" color (absorption of light 1.57 per cent per cm.) was later found to contain 0.080 per cent total iron as Fe_2O_3. In the first case the glass was noticeably green with a bluish tinge; in the latter glass the color was not nearly so green.

Such results may be an indication that there is considerably less dissociation in the case of boron glasses than in barium glasses. Similarly there may well be large differences in the effect on the dissociation of ferric oxide dissolved in glasses containing sodium on one hand and potassium on the other, thus accounting in part for the better color of potash glasses and the fact that when manganese dioxide is used as a decolorizer less of it is necessary with potash than with soda glasses. If this decolorizer acts primarily as an oxidizing agent the smaller quantity required with potash glass would indicate less dissociation than with soda glasses.[1]

[1] Potash glass made from raw materials containing the same amount of iron as the corresponding soda glass might still show less iron than the soda glass because the higher fluidity of the latter glass at high temperatures would presumably cause more pot corrosion and correspondingly greater iron absorption.

Since the amount of dissociation increases with temperature it is obvious that the higher the temperature at which an iron bearing glass is fined the greener the color. And also it follows that more decolorizer is required for a glass fined at the elevated temperature than at a lower temperature. Of course, other factors such as pot attack and volatilization (or dissociation) of decolorizer are also increased as the temperature is raised, but added to these is the fact demonstrated in this paper that dissociation of the iron oxide increases with increasing temperature of the glass.

Incidentally, it may be noted here that the foregoing data furnish the basis for an additional explanation of the phenomenon noted by Washburn[1] that the color of optical glass increases after stirring is started. His theory was, briefly, that iron or iron oxide volatilized or carried mechanically from the burners of the furnace, was absorbed by the glass[2] from the furnace atmosphere during the stirring process. The demonstration of the present paper indicates another factor that plays a part in the intensification of color observed by Washburn, namely, dissociation of the iron oxide already present in the glass.

While dissociation of the iron oxide present in the glass would account for some intensification of color after stirring was started, there is another important factor giving the same effect,—the mixing, by the motion of the stirrer, of the more basic layer on the bottom of the pot with the other portions of the glass. One observation made at the plant of the Bausch and Lomb Optical Company may be cited here in support of this explanation. A proof taken from a melt of dense flint (n_D 1.65) before the glass was fine gave an iron content of 0.019 per cent Fe_2O_3. This sample was very light in color and from its index of refraction F. E. Wright estimated it to contain 46 per cent PbO. The final glass, however, showed a very much deeper color and from its index of refraction it was estimated to contain 51 per cent

[1] THIS JOURNAL, **1**, 637 (1918).

[2] Iron salts in general are not very volatile. In previous work at the Geophysical Laboratory in which ferric oxide was heated to higher temperatures than those found in glass furnaces no evidence of volatilization of ferric oxide was noted.

PbO and analysis showed no less than 0.062 per cent Fe_2O_3. From these observations it is quite obvious that the lead oxide had segregated during melting and had formed a lead-rich layer on the bottom of the pot. This layer was very corrosive and had attacked the pot, dissolving therefrom a certain amount of iron. Consequently when this heavy layer was stirred up through the rest of the glass the color was increased not by absorption of iron from the furnace gases, but simply by mixing the iron-carrying lower layer with the bulk of the glass.

GEOPHYSICAL LABORATORY
CARNEGIE INSTITUTION OF WASHINGTON

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[1] The abbreviation (*C. A.*) at the end of an abstract indicates that the abstract was secured from the Editor of *Chemical Abstracts* by coöperative agreement.

General and Miscellaneous

1. The manufacture of ultramarine blue. M. SAUVAGEOT. *Rev. prod. chim.*, **24,** 141–8, 205–12(1921).—General description of the raw materials employed and of the method of manuf. of ultramarine blue, together with tables giving 22 analyses of blues and 3 analyses of ultramarine green and a brief mention of some of the modern theories on the constitution of ultramarine and of the improvements which should be effected in the mfg. process. A. P. C. (*C. A.*)

2. The industrial preparation and filtration of colloidal solutions and masses. J. H. FRYDLENDER. *Rev. prod. chim.*, **24,** 173–82(1921).—The main principles of colloidal chemistry are very briefly reviewed. Plauson and Block's "colloidogenetic" mill is described together with its application to various industries including fuller's earth, fuel (liquid coal), graphic lubricants, and ultramarine. Plauson's method of ultrafiltration (see Schmitt, *C. A.*, **14,** 3763) is described and mention is made of its applications in the industries of *ceramics*, mineral pigments, and dust prevention. A. P. C. (*C. A.*)

3. The measurement of color. C. E. K. MEES. Eastman Kodak Co. *J. Ind. Eng. Chem.*, **13,** 729–31(1921).—Methods of color measurement are discussed and the spectroscope, spectrometer, colorimeter, monochromatic analyser, trichromatic analyser, and the Eastman colorimeter described. The last is robust in construction and uses three colored wedges, each wedge absorbing $^1/_3$ of the spectrum. With these wedges and a fourth grey wedge any color can be perfectly matched and quantitatively measured. The instrument is especially useful for the rapid and quantitative measurement of the color of *glasses, porcelains,* and *enameled* surfaces. Ed.

4. Clays and earths. In the tariff act of 1913. U. S. TARIFF COMMISSION. *Tariff Information Surveys,* **1921.** 5c a copy from Supt. of Documents, Wash. D. C.—**Kaolin or China clay:** summary: *General Information* on tariff classification, descrip., uses, domestic production, geographical distribution, ball clay, equipment, methods of manuf., organization, domestic production and consumption, domestic exports, foreign production, imports and revenue, costs and prices, tariff history, competitive conditions, tariff considerations; *Production in the U. S.*, states, (kaolin and paper clay) and principal countries; imports by countries, imports for consumption, prices, rates of duty, court and treasury decisions.

Clays other than kaolin or China clay: summary; ball clay; common blue clay and all other clays; general information on tariff classification, description and uses, domestic production, foreign production, imports, prices, tariff history, competitive conditions, tariff considerations; domestic production of ball clay; imports by countries; imports for consumption, domestic exports; prices; rates of duty; court and treasury decisions.

Fuller's earth: summary; general information on tariff classification, description, uses, domestic production, foreign production, imports, prices, tariff history, competitive conditions; domestic production; imports for consumption; prices; rates of duty.

Fluorspar: summary of description, domestic production, foreign production, competitive conditions, imports, prices, tariff history, duties in foreign countries, tariff questions involved; general information on description, grades, fluorspar for optical purposes, uses, occurrence, deposits in the U. S., foreign deposits, preparation, production, imports, markets and prices, consumption; production in the U. S., states; production in principal foreign countries; imports by countries; imports for consumption, calendar years; imports for consumption, fiscal years; prices, rates of duty; competitive conditions; court and treasury decisions.

Feldspar, crude and manufactured: general information on tariff classification, description, uses, domestic production, foreign production, imports, prices, tariff history, competitive conditions; production in the U. S.; production in foreign countries; rates of duty; court and treasury decisions. Ed.

5. New Ceramic School in Canada. ANON. *Chem. Met. Eng.*, **25,** 209 (1921).—A ceramic department is to be established in the University of Saskatchewan, Saskatoon, Can. W. G. Worcester, a graduate of the Ohio State University Ceramic Department, has been appointed professor in the new department. H. F. S.

6. Molecular force and plasticity of clays. HERBERT CHATLEY. *Trans. Ceram. Soc.* (Eng.), **19,** 1–2(1919–20).—Suggestion is made that plasticity (and also, when the molecules of the fluid are considered, the specific "colloid" properties of sub-divided matter) is due to the presence of molecular forces comparable with weight or other external agency. H. F. S.

PATENTS

7. Silicate containing materials of fat-like character and high absorptive power. ROBERT MARCUS. Ger. 322,088, Aug. 22, 1917. Sol. silicates are made to interact with hydroxy acids in suitable ratios. As hydroxy acids, lactic acid, glycolic acid or the like may be employed. The resulting salts remain emulsified in the state of finest subdivision with the colloidal silicic acid. They prevent the conversion of the silicic acid to crystalline form and are the cause of the cleansing power of the mass. The mass is used as a *substitute for soap.* It is claimed to be serviceable also as *lubricant.* (*C. A.*)

BOOK

8. Sprechsaal-Kalender für die Keramischen, Glas-ünd verwandten Industrien. 1921. Edited by J. KOERNER. Coburg: Müller & Schmidt. M 7.50. For review see *Tonind.-Ztg.*, **45,** 464 (1921). (*C. A.*)

Apparatus and Instruments

9. Suggestions for calibrating base metal thermocouples by the freezing point method. KIRTLAND MARSH. *Chem. Met. Eng.*, **24,** 1071(1921).—Iron protection tubes can be used in molten metallic tin and aluminum without contaminating the metals, if the tubes are heated to 900°F and then dipped into a wash of one part plumbago and two parts water. For use in molten copper a second dipping is given into a wash of one part lime and one part water. H. F. S.

10. Electrical pyrometry. ANON. *Elektrochem. Z.*, **27**, 46, 52, 63, 72, 81, 85(1920–21). C. G. F. (*C. A.*)

11. Drying theory; most economical temperatures; graphic determination of moist air. E. HOEN. *Z. Ver. deut. Ing.*, **63**, 821–6(1919); *Mech. Eng.*, **42**, 169–71(1920); *Science Abstracts*, **23B**, 561–2.—A discussion of the most economical temp. and most economical degree of satn. for the air at the outlet from the drying chambers; discussion of influence of air pressure; and graphical methods for the solution of problems in connection with processes of drying. The conditions most easily attainable mechanically and still satisfactory from an economic point of view lie, so far as temp. is concerned, between 60 and 80 and 60 to 80% of satn. In this case we expend roughly 1 kg.-cal. of heat to take care of 1.4 g. of water, which means an expenditure of 0.7 kg.-cal. of heat for 1 g. of water, or 700 kg.-cals. of heat for 1 kg. of water. This is very nearly the same as the heat necessary to evap. 1 kg. of water at atm. pressure, which is 640 kg.-cals. Under this condition the efficiency of drying is close to 90%. H. G. (*C. A.*)

BOOKS

12. BÜHLER, F. A.: **Filtern und Pressen, zum Trennen von Flüssigkeiten und festen Stoffen.** 2nd Ed. revised by Ernest Jänecke. Leipzig: Otto Spamer. 172 pp. M 37. For review see *Chem. Weekblad*, **18**, 224 (1921). (*C. A.*)

Chemistry, Physics, and Geology of Raw Materials

13. The colorimetric determination of iron in silicates with ammonium thiocyanate. JOSEF MATEJKA. *Chem. Listy*, **15**, 8–13(1921).—Thomson (*J. Chem. Soc.*, **47**, 493(1895)) and Proskauer made use of NH_4CNS for the colorimetric detn. of Fe. The procedure adopted by M. is as follows: Into a 20-cc. colorimetric tube place 1–2 cc. of standard Fe soln. (a soln. of $NH_4Fe(SO_4)_2.12H_2O$ in 0.5% H_2SO_4 giving a concn. of 0.01 g. of Fe_2O_3 per l.), 8 cc. of a mixt. consisting of 1 part of 10% HNO_3 and 3 parts of 10% citric acid by vol., and then add 8 cc. of a 10% soln. of NH_4CNS. Dil. the mixt. to the 20 cc. mark. To two other tubes add the same solns. with the exception of the one containing the Fe and dil. to a vol. of about 19 cc. To one of these add drop by drop until the intensity of coloration is the same as in the standard tube, the soln. of the sample to be tested obtained by dissolving its pyrosulfate fusion in 0.5% H_2SO_4 and dilg. to 250 cc. Then make a second detn., taking care to have the final vol. in the colorimetric tube exactly 20 cc. The following precautions are to be observed: The thiocyanate must be added in excess and its concn. must be the same in both tubes (cf. Tatlock, *Z. anal. Chem.*, **28**, 706(1889)). K_2SO_4 and $KHSO_4$ cause a reduction of the intensity and change the color from pink to orange. NaCl, $MgCl_2$, $CaCl_2$, $TiCl_3$ and Al salts have a detrimental effect. The interference of all these salts is inappreciable if the soln. to be tested is sufficiently acidified with HNO_3 and citric acids before the addition of thiocyanate. The concn. of the Fe in the

soln. in the tube should be between 0.00001 and 0.00002 g. Fe_2O_3. The reagents should be absolutely free from Fe. M. has found the above methods as satisfactory as the ether method of Lunge (*Z. angew. Chem.*, **1886**, 3).

JOHN M. KRNO (*C. A.*)

14. The composition of dolomites. IRENE EVERMAN, O'NEAL MASON, AND GLENN BROWNING. *Chem. News*, **122**, 109–10(1921).—Thirteen analyses of dolomites from the following localities are given: Le Grand, Iowa; Gouverneur, N. Y.; Cape Breton Island; Frizington, England; Marquette, Mich.; Tuckahoe, N. Y.; New Almaden, Calif.; Providence, R. I.; White Pine, Nev.; Guanajuato, Mex.; Nagyag, Transylvania; Clayton, Iowa; and Dubuque, Iowa. S. G. GORDON (*C. A.*)

PATENT

15. Alumina from clay. WILHELM DAN BERGMAN. Norw. 30,846, June 28, 1920. Clay is decomposed with an acid soln. and the $Al(OH_3)$ pptd. by means of an alk. soln. for which purpose the solns. forming in the electrolysis of a neutral sulfate soln. are used. From the acid anode liquid the neutral sulfate is sepd. by crystn., said liquid then being used to decompose the clay. The resultant Al soln. is sepd. from the insol. constituent (silicic acid) and pptd. with the alk. cathode liquid. $Al(OH)_3$ then seps. off and neutral sulfate re-forms, which is again returned to the process. (*C. A.*)

Refractories and Furnaces

16. Judging sands for foundry use. HENRY B. HANLEY AND HERBERT R. SIMONDS. *Foundry*, **48**, 772–4, 867–8, 875, 921–2(1921).—Sand for foundry use has 3 characteristics: refractoriness, cohesion and porosity. Foundry sands may be divided into molding sand, core sand, facing sand, fire sand, gravel, high-silica sand and parting sand. Molding sand is usually a natural sand and a mixt. of quartz, feldspar and clay. Core sand is an artificial sand containing a binder as molasses. Chem. analyses of typical sands are given and their interpretation is discussed. Other tests for sand are also discussed. The best test for the amount of bond in a sand is the adsorption of methyl violet. This is best detd. by adding the dye to a soln. of the colloids from the sand till a color is produced and then removing the dye by means of mordanted cotton yarn and comparing the color of the yarn with that of standards. The strength of the bond depends on the surface of the sand particles as well as on the amt. of bond. Methods of chem. and mechanical analysis are given in detail. R. S. DEAN (*C. A.*)

17. New German refractory product. ANON. *Chem. Met. Eng.*, **24**, 1070(1921).—A coating composed of 75% carborundum and 25% sodium silicate is painted on to fire bricks. The layer is about one fiftieth of an inch thick and fuses to a glassy veneer which is said to be remarkably resistant to mechanical injury and the chem. action of flames. It has been used successfully as a lining for gas retorts. H. F. S.

18. Refractories for bottom-connected electric furnaces. F. W. BROOKE. *Iron Age*, **106**, 1316(1920).—Successful operation of elec. furnaces having a conductive bottom depends upon: correct application of the current, the use of good double-burned *dolomite* and a good pitch or tar, and good operators. The transformer connected directly to the bottom is designed to carry a heavier current than that carried by each electrode, which necessitates the bottom transformer having such a kw.-amp. capacity and a secondary voltage that when the top electrodes carry equal currents there is a perfect balance on the primary side. These features insure carriage of all current given to the furnace, no elec. trouble from the deep holes formed during melting, and long life of furnace linings. A good pitch consists of the residue from coal tar distn. up to 250°. Quick "coming on" of the conductivity is effected by designing the furnace and putting in the bottom in such a way that all the excess tar will quickly drain away. W. H. BOYNTON (*C. A.*)

19. Quartzites and silica bricks. F. WERNICKE. *Stahl u. Eisen*, **40**, 432–7(1920).—For the manuf. of silica bricks in Germany, rocks of 3 different geological formations are employed, *viz.*, felsquartzites—marine deposits belonging to the Devonian period—which have a dense structure of closely interlocked crystals, the individual quartz grains being relatively large, with sharply defined edges and a characteristic wavy extinction of color. Carboniferous sandstones, consisting of medium-sized quartz crystals, some having sharply defined edges and closely interlocked, while in others the edges are corroded and the crystals are sepd. by a small quantity of cement-like material; and glacial or "amorphous" quartzites—fresh water deposits of Tertiary origin belonging to the period of lignite formation—consisting of very small grains of quartz with rounded edges distributed uniformly through an amorphous ground-mass of siliceous gel or "basaltic" cement. All these quartzites are very rich in silica; their refractoriness corresponds to that of cone 36 (1790°). In the manuf. of silica bricks it is not necessary to effect a complete conversion of quartz into tridymite provided the max. expansion is attained. With glacial quartzites this is reached by one firing at 1450° without excessively prolonged heating but silica rocks from the coal measures require 2 or 3 firings before they attain their max. expansion, and felsquarzites continue to expand after several prolonged firings. By examn. under the microscope with a magnification of only 45 diams. using polarized light, useful quartzites can be distinguished from the useless ones, as the former contain minute crystals in a glassy ground-mass, while the latter consist almost wholly of larger crystals or sharply defined fragments of such crystals. For the arches of elec. and other metallurgical furnaces, silica bricks should be made of glacial or Tertiary quartzites. For glass-melting furnaces, where the temp. is not so high, silica bricks made from coal measure quartzites are satisfactory, and may be recommended on account of their greater purity and the lesser risk of spoiling the glass. Coal measure quartzites may also be used for silica bricks employed in regenerators, coke ovens, etc. Mixts. of Tertiary and coal measure quartzites are also used. Coarse cryst. quartzites are only suitable for low-grade silica bricks used in annealing furnaces, and so forth. (*C. A.*)

20. Laboratory furnace for deformation tests of refractories. L. R. OFFICE. *Chem. Met. Eng.*, **25,** 162(1921).—The furnace consists of an ordinary pot furnace with one burner, set tangentially to the inner surface. Natural gas and compressed air at 30 pounds pressure are used. For high temp. the air is preheated by being passed through a heater consisting of four two-foot lengths of two inch iron pipe connected at the ends with U's and spaced so that the flame from two gas burners will surround them. The air is raised to a temp. of 250°C at the burner and cone 35 can be reached in the furnace in 35 min. H. F. S.

PATENTS

21. Refractory articles. O. REBUFFAT. Brit. 159,865, Jan. 5, 1921. Bricks and other refractory articles composed chiefly of SiO_2 are made from a mixt. contg. a small proportion, *e. g.*, 0.45% of phosphoric, tungstic, molybdic, boric or other acid stable at high temps., or a salt of such acid. The bricks are baked at a temp. of 1,300–1,500° to convert the SiO_2 into modifications of low sp. gr., such as tridymite. (*C. A.*)

22. Refractory substances. OSMOSIS CO., LTD., D. NORTHALL-LAURIE AND W. R. ORMANDY. Brit. 159,537, Jan. 22, 1920. Refractory articles are made by firing specially purified china clay without a flux at temps. below 1500°. The clay is purified by the methods set forth in 2379, **1911** (*C. A.*, **6,** 1964), 3364, **1911**(*C. A.*, **6,** 2036), 27,930, **1911** (*C. A.*, **7,** 1846), 27,931, **1911** (*C. A.*, **7,** 1846), 28,185, **1911** (*C. A.*, **7,** 1846), and 14,235, **1912** (*C. A.*, **7,** 4055), *e. g.*, it is made into a slip with a dil. electrolyte such as NaOH or Na_2SiO_3, allowed to stand for about 24 hrs., and is either subjected to electroösmosis, or pptd. by a coagulating agent such as $Al_2(SO_4)_3$ and reconverted to the sol. condition by a treatment with an alkali. The purified clay is made unto a slip to which a grog made of vitrified china clay may be added to minimize contraction on firing. (*C. A.*)

BOOK

23. Special Reports on the Mineral Resources of Great Britain. XIV. Refractory Materials: Fireclays. Memoirs of the Geological Survey. London: H. M. Stationery Office. 243 pp. 8s. (*C. A.*)

Abrasives

24. Abrasive materials. In the tariff act of 1913. U. S. TARIFF COMMISSION. *Tariff Information Surveys*, **1921.** 5c a copy from the Supt. of Documents, Wash., D. C.—This bulletin deals with the description of abrasive materials, their method of manuf., list of mfgrs., production, consumption, and imports. The statistics regarding domestic production are classified by states and by kinds; those regarding imports, by countries and by kinds. Prices, rates of duty, and court and treasury decisions on tariff classifications and rates are also covered. Ed.

White Ware and Porcelains

See Abst. Nos. 3 and 4.

Glass

25. Quartz glass and the mercury lamp. G. BERLEMONT. *Bull. soc. encour. ind. nat.*, **133,** 254–60(1921); 2 figs.—During the war B. was induced to resume his attempts to perfect the manuf. of the mercury vapor lamp. In collaboration with Henri George and others he succeeded in making a lamp for medical and experimental uses. Tungsten was employed for sealing with the quartz glass and the arc was not formed in a vacuum but at atmospheric pressure, thereby attaining two improvements: making the lamp more robust and simplifying the lighting procedure. J. B. PATCH (*C. A.*)

26. Canadian glass industry in 1918. ANON. *Chem. Met. Eng.*, **25,** 115(1921).—Nine glass factories were in operation. Six made lamp chimneys, lantern globes, bottles and other pressed and blown ware; two made vials and chemical glassware; one made sheet glass. The total production amounted to $6,578,000. Of this $35,000 was exported. During the same year $5,480,000 worth of glassware was imported. H. F. S.

27. Electrically heated glass-annealing lehrs. E. F. COLLINS. *Chem. Met. Eng.*, **25,** 119.—See THIS JOURNAL, **4,** 600(1921). H. F. S.

PATENT

28. Light filters. E. L. FRIEDMANN & Co. Brit. 159,179, Feb. 4, 1921. Filters for lamp-shades, etc., for producing artificial daylight by gas-filled metallic filament lamps consist of glass in which cobalt-blue and nickel-green colors are melted together in one charge. Other additions such as $CuSO_4$ may be used for the production of a green color. (*C. A.*)

Enamels

29. Electric vitreous enameling furnace. ANON. *Chem. Met. Eng.*, **25,** 206. See THIS JOURNAL, **4,** June (1921). H. F. S.

30. Electric heat raises enameling output. J. W. CARPENTER. *Elec. World*, **77,** 873–5(1921).—See THIS JOURNAL, **4** (1921). W. H. BOYNTON (*C. A.*)

See also Abst. No. 3.

Cement, Lime and Plaster

31. Lime, gypsum, and cements. In the tariff act of 1913. U. S. TARIFF COMMISSION. *Tariff Information Surveys*, **1921.** 5c a copy from Supt. of Documents, Wash., D. C.—**Lime:** summary; *General information* on description, important uses, domestic production (raw materials, process of manuf., organization, geographical distribution, domestic productions, exports), imports, tariff history, competitive conditions (general situation, competition in Wash. district, Canad. duty on lime); *Production in the U. S.*, by states, by uses, total lime burned and sold and hydrated lime (since 1906, quantity,

value, average price, number of plants); imports by countries; imports by consumption, since 1907; domestic exports, since 1909; rates of duty, court and treasury decisions.

Gypsum, crude, and calcined, and manufacture of: summary; general information on tariff classification, description and uses, domestic production (raw material, equipment, methods of manuf., organization, geographical distribution, history of the industry, domestic production and consumption, domestic exports); foreign production; imports; prices; tariff history; competitive conditions; tariff considerations; production in the U. S. of gypsum, and manuf. of; production in principal countries; imports by countries of plaster rock or gypsum (crude, ground or calcined, crude and ground or calcined, manuf. of), cement, Keene's cement; imports for consumption of plaster rock or gypsum, crude, ground or calcined; imports for consumption of cement, Keene's cement and other plaster rock or gypsum, plaster of Paris, and so forth; domestic exports, prices, rates of duty, court and treasury decisions.

Cement, Roman, Portland, and Other Hydraulic: summary; general information on tariff classification, description, uses, military importance, domestic production of raw materials, equipment, mfg., methods, organization, geographical distribution, history of the industry, domestic production, and consumption, domestic exports, foreign production, imports, prices, tariff history, competitive conditions, tariff considerations; production in the U. S., imports by countries; imports by consumption; domestic exports; average factory price per barrel; rates of duty; court and treasury decisions.

ED.

32. Specifications for the color of gypsum plasters. W. E. EMLEY AND C. H. FAXON. *Chem. Met. Eng.*, **24,** 1054–5(1921).—Advocates the use of spectrophotometric examination and grading. H. F. S.

33. Ferroportland pozzuolana mixtures. FABIO FERRARI. *Giorn. chim. ind. applicata*, **2,** 549–54(1920).—F. comes to the following conclusions from expts.: (1) Normal agglomerates made up of portland-pozzuolana mixts. show, in general, an imcomparably greater resistance to the action of sulfates and chlorides than pure portland agglomerates, owing to the lower sp. permeability of the 1st type and its lower content in Ca aluminates. They become, therefore, passive after a longer or shorter period of time to the disintegration to which all mortars are subjected that contain strongly basic aluminates, as to alterations in their superficial continuity (the essential coeff. of mechanical resistance of every cementitious mass constituted of hydrated Ca silicates). (2) Normal agglomerates constituted of ferroportland (or portland free of binary Fe and Al compds.), in that they are free from defects derived from the presence of aluminates, undergo during long periods of time a regression because of the diffusion of CaO due to their high sp. permeability and the instability of their protective crust, characters with which they are endowed because of the high basicity of ferroportland. (3) Mortars constituted of ferroportland-pozzuolana mixts., because of the

absence of both the defects mentioned above, present an abs. stability of their protective stratum and a practically perfect impermeability. Such mixts. are, therefore, the only ones rationally applicable at present to maritime construction or works intended to come into contact with sulfated waters.

ROBERT S. POSMONTIER (*C. A.*)

34. Influence of additions of calcium sulfate or chloride on the shrinkage of cement. A. GUTTMAN. *Zement*, **9**, 310–3, 429–32(1920).—Addition of raw gypsum or $CaCl_2$ to portland, iron-portland, or blast-furnace cements cause them to swell so that the natural shrinkage is counteracted or only takes place after a long time. Cements so treated are especially adapted for use as mortars where it is desired that the joint should be nearly invisible.

(*C. A.*)

PATENT

35. Burning lime. N. WOLLAN AND KARL SANDE. Norw. 30,821, June 14(1920). Finely divided limestone is made to fall freely through gas heated to a high temp. (*C. A.*)

BOOK

36. Zehn Jahre Portlandzement Prüfung. HANISCH, AUGUST. Leipzig: Arthur Felix, 31 pp. M. 5. For review see *Z. angew. Chem.*, **34**, Aufsatzteil, 159(1921). (*C. A.*)

ACTIVITIES OF THE SOCIETY

New Members Received during September and October

Associate

Bales, Cecil E., Louisville Fire Brick Works, Highland Park, Ky.
Burt, N. P., Great Western Stove Co., Leavenworth, Kan.
Chesler, Isidor, 710 East 14th St., New York City.
Cole, Sanford S., Alfred, N. Y.
Doane, G. Earl, Poplar Bluff, Mo.
Handy, James O., Pittsburgh Test Laboratory, Box 115, Pittsburgh, Pa.
Jackson, Wynne L., 111 S. Kilpatrick Ave., Chicago, Ill.
Krak, J. B., 42 Hammond St., Jamaica, N. Y.
Lynch, Richard H., 8th and O'Donnell Sts., Baltimore, Md.
Montgomery, R. A., Springfield Paving Brick Co., Springfield, Ill.
Quay, Paul Q., 15620 Euclid Ave., East Cleveland, Ohio.
Rennieburgh, Cedric L., 625 W. Main St., Zanesville, Ohio.
Rodgers, Louis E., 30 N. LaSalle St., Chicago, Ill.
Rushmore, Murray, 120 West 8th St., Plainfield, N. J.
Stamm, Charles L., 212 W. 2nd St., Mt. Vernon, N. Y.
Wright, M. H., Tennessee Enamel Mfg. Company, Park Ave. and Railroad, West Nashville, Tenn.

Foreign Associate

Kanashima, Shigeta, Tokio Technical College, Asakusa, Tokio, Japan.
Maruyama, Junkichi, 7 Icchome, Gojotori, Chikko, Osaka, Japan.

Corporation

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U. S. Metals Refining Co., Chrome, N. J.

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B. F. Drakenfeld & Company, Inc.
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East Liverpool Potteries Company
Edgar Plastic Kaolin Company
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Gillinder Brothers
Gleason-Tiebout Glass Company
Golding Sons Company

Hall China Company
Hanovia Chemical & Mfg. Company
Harbison Walker Refractories Company
Harker Pottery Company
Harshaw, Fuller & Goodwin Company
Homer-Laughlin China Company
L. J. Houze Convex Glass Company

Illinois Glass Company

Jefferson Glass Company
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Johnston Brokerage Company
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Matawan Tile Company
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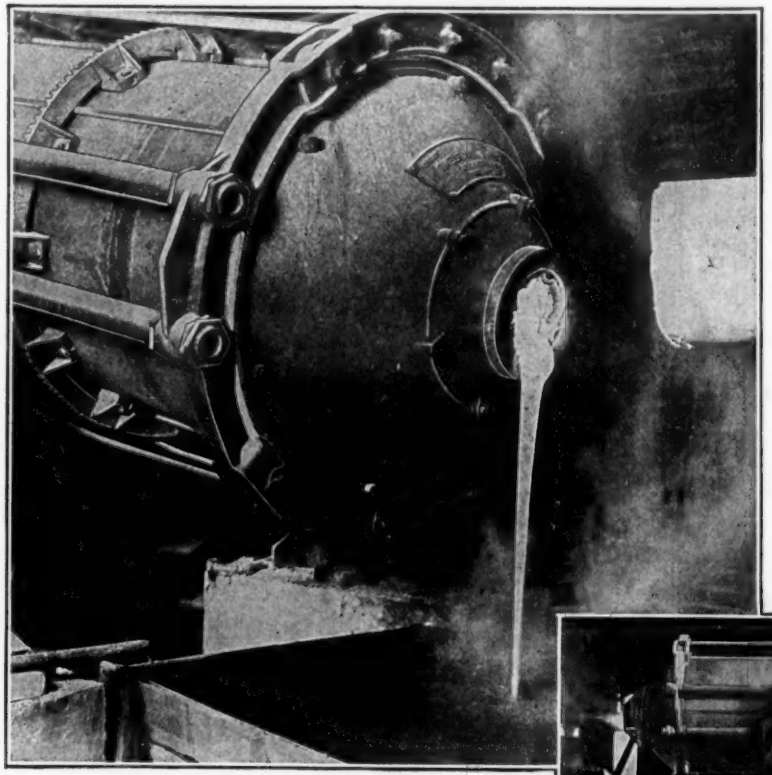

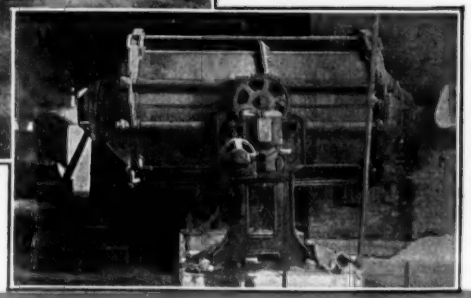

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Goebel & Co., J.
Paper Makers Chemical Co.
Roessler & Hasslacher Chemical Co.
Clay (Electrical —Porcelain)
Edgar Brothers Co.
Johnson Porter Clay Co.
Paper Makers Chemical Co.
Clays (Enamel)
Goebel & Co., J.
Clay (Fire)
Edgar Brothers Co.
Goebel & Co., J.
Paper Makers Chemical Co.
Grand View Fire Clay Mines
Clay (Pot, Tank Blocks)
Goebel & Co., J.
Clay (Potters)
Goebel & Co., J.
Clay (Sagger)
Edgar Brothers Co.
Grand View Fire Clay Mines
Johnson Porter Clay Co.
Paper Makers Chemical Co.
Clay Handling Machinery
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.
Clay Miners
Edgar Brothers Co.
Grand View Fire Clay Mines
Johnson Porter Clay Co.
Clay Washing Machinery
Crossley Machine Co.
Mueller Machine Co., Inc.
Clay Working Machinery
Abbé Engineering Co.
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.
Colors
Drakenfeld and Co., B. F.
Harshaw, Fuller and Goodwin Co.
Roessler and Hasslacher Chemical Co.
Vitro Mfg. Co.
Conditioning Machinery
Philadelphia Drying Machinery Co.
Conical Mills
Hardinge Co.
Conveyors (Clay, Sand, Brick, etc.)
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Philadelphia Drying Machinery Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.
Controllers (Automatic Temperatures)
Charles Engelhard
Controllers (Electric)
General Electric Co.
Crucibles (Clay, Sand & Black Lead)
Goebel & Co., J.
Crushers
Abbé Engineering Co.
Chambers Brothers Co.
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Hardinge Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.
Decorating Supplies
Drakenfeld and Co., B. F.
Harshaw, Fuller and Goodwin Co.
Roessler and Hasslacher Chemical Co.
Vitro Mfg. Co.
Disintegrators
Abbé Engineering Co.
Chambers Brothers Co.
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.
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Manufacturers Equipment Co.
Dryers (China Ware—Porcelain)
Philadelphia Drying Machinery Co.
Proctor and Schwartz, Inc.
Russel Engineering Co.
Dryers (Radiated Heat)
Manufacturers Equipment Co.
Drying Machinery
Crossley Machine Co.
Philadelphia Drying Machinery Co.
Proctor and Schwartz, Inc.
Manufacturers Equipment Co.
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Wilson-Maeulen Co.
Electrical Porcelain Machinery
Crossley Machine Co.
Mueller Machine Co., Inc.
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Enameling Furnaces
The Porcelain Enamel & Mfg. Co.
U. S. Smelting Furnace Co.
Enameling Muffles
Parker-Russell Mining & Mfg. Co.
Enameling, Practice Service
The Porcelain Enamel & Mfg. Co.
Enamels, Porcelain
The Porcelain Enamel & Mfg. Co.
Engineering Service
Abbé Engineering Co.
Crossley Machine Co.
Chambers Brothers Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.

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The highest grade of Silica from North Carolina is pulverized in the Hardinge Conical Mill. The product is of uniform fineness and pure.

If you have a problem of grinding any hard or abrasive material, the results of grinding this hard silica, will give you an idea of what can be accomplished.

Material	Hard, abrasive Silica
Size of feed to mill	¾"
Size of product	90% passing 200-mesh Screen
Capacity	1 ton per hour
Mill	6' x 48" Hardinge Conical Pebble Mill, equipped with air separator.
R.P.M. of mill	30
H.P. input	40 (Total)
Life of Lining	8 years (approximately)

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Chambers Brothers Co.

Feldspar
Drakenfeld and Co., B. F.
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O'Brien and Fowler
Roessler & Hasslacher Chemical Co.

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Crossley Machine Co.
Mueller Machine Co., Inc.

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Furnaces
U. S. Smelting Furnace Co.
Parker-Russell Mining & Mfg. Co.
The Porcelain Enamel & Mfg. Co.
The Surface Combustion Co.

Gold
Harshaw, Fuller and Goodwin Co.
Roessler and Hasslacher Chemical Co.

Glazes and Enamels
Harshaw, Fuller and Goodwin Co.
Roessler and Hasslacher Chemical Co.
Vitro Manufacturing Co.

Ilmenite
Buckman and Pritchard, Inc.

Impervite (Refractory and Hard Porcelain)
Charles Engelhard

Jar Mills
Abbé Engineering Co.

Jiggers
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Mueller Machine Co., Inc.

Kaolin
Edgar Plastic Kaolin Co.
Harshaw, Fuller and Goodwin Co.
Roessler & Hasslacher Chemical Co.

Kryolith
Pennsylvania Salt Mfg. Co.

Laboratory Mills
Abbé Engineering Co.
Crossley Machine Co.

Magnesia Refractories
General Ceramics Co.

Mills (*See under Ball Mills*)
(*See under Pebble Mills*)

Minerals
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Harshaw, Fuller and Goodwin Co.
Roessler and Hasslacher Chemical Co.

Mixing Machines
Chambers Brothers Co.

Muriatic Acid
Harshaw, Fuller and Goodwin Co.
Pennsylvania Salt Mfg. Co.
Roessler and Hasslacher Chemical Co.

Oxides
Drakenfeld and Co., B. F.
Harshaw, Fuller and Goodwin Co.
Paper Makers Chemical Co.
Pennsylvania Salt Mfg. Co.
Roessler and Hasslacher Chemical Co.

Pans (Wet and Dry)
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Crossley Machine Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.

Pebble Mills
Abbé Engineering Co.
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Hardinge Co.
Mueller Machine Co., Inc.

Plate Feeders
Chambers Brothers Co.
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.

Porcelain Balls and Lining
Abbé Engineering Co.

Porcelain Enameling Plants and Equipment
The Porcelain Enamel & Mfg. Co.

Porcelain Enameling Service, Practical
The Porcelain Enamel & Mfg. Co.

Porcelain Enamels
The Porcelain Enamel & Mfg. Co.

Pottery Machinery
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Crossley Machine Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.

Producer Gas Burning Systems
Manufacturers Equipment Co.

Pug Mills
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Crossley Machine Co.
Hadfield-Penfield Steel Co.
Manufacturers Equipment Co.

Pulverizing Machinery
Abbé Engineering Co.
Crossley Machine Co.
Hadfield-Penfield Steel Co.
Hardinge Co.
Manufacturers Equipment Co.
Mueller Machine Co., Inc.

Pulverizing Mills
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Crossley Machine Co.
Hadfield-Penfield Steel Co.
Hardinge Co.
Mueller Machine Co., Inc.

Pumps
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Mueller Machine Co., Inc.

Pyrometers (Indicating)
Charles Engelhard
Wilson-Maeulen Co.

Pyrometers (Recording)
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Wilson-Maeulen Co.

Pyrometer Tubes (Refractory and Hard Porcelain)
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Montgomery Porcelain Products Co.

Recording Instruments
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Refractory Materials
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Parker-Russell Mining & Mfg. Co.

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Crossley Machine Co.
Hadfield-Penfield Steel Co.
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Drakenfeld and Co., B. F.
Vitro Mfg. Co.

Silica Brick
Parker-Russell Mining & Mfg. Co.

Silica (Fused)
General Ceramics Co.

Silex Lining
Abbé Engineering Co.
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Smelters
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Charles Engelhard
Wilson-Maeulen Co.
Tile Machinery (Floor and Wall)
Crossley Machine Co.
Mueller Machine Co., Inc.
Tubes (Insulating)
Montgomery Porcelain Products Co.
Tubes (Pyrometer)
Charles Engelhard
Montgomery Porcelain Products Co.
Tube Mills
Abbé Engineering Co.
Tunnel Kilns
Russell Engineering Co.
Vacuum Pumps
Abbé Engineering Co.
Whiting
Drakenfeld and Co., B. F.
Harshaw, Fuller and Goodwin Co.
Roessler and Hasslacher Chemical Co.
Zircon (Miners of)
Buckman and Pritchard, Inc.
Zirconium Silicate (Fire Cement)
Buckman and Pritchard, Inc.
Zirconium Silicate (Refined)
Buckman and Pritchard, Inc.

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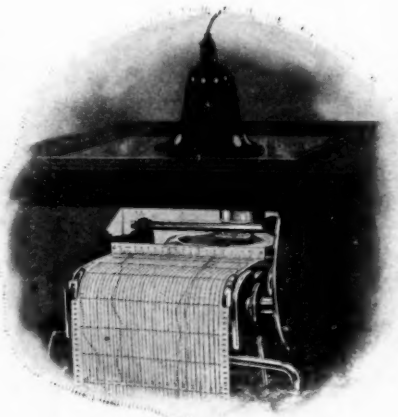

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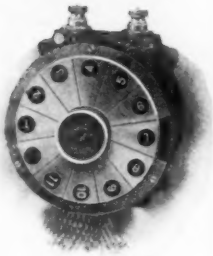

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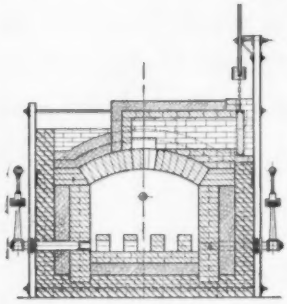

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How The JOURNAL Produces

Nov. 4, 1921

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We are in the market for an enameled smelter, and have written to the manufacturers whose advertisements appear in the Journal.

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Engelhard Pyrometers Are Good Pyrometers To Standardize On.

Charles Engelhard, Inc.

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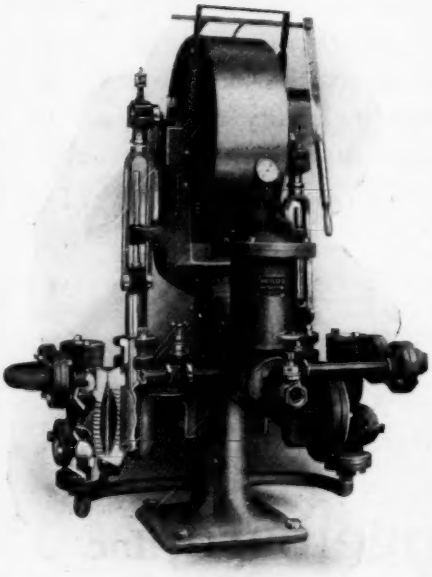

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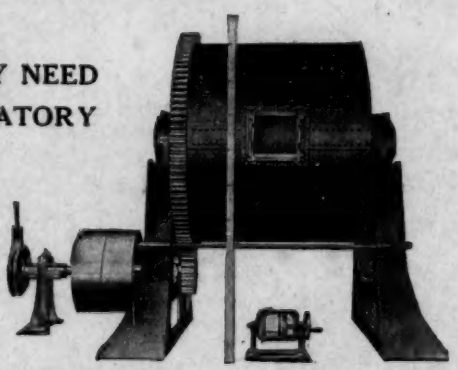

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